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NPL REPORT MAT 102

MECHANICAL TESTING OF FIBRE-REINFORCED POLYMER MATRIX COMPOSITES AT CRYOGENIC TEMPERATURES

Requirements for mechanical test capability at -269°C (4 K)

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MARCH 2022

Mechanical Testing of Fibre-Reinforced Polymer Matrix Composites at Cryogenic Temperatures:
Requirements for Mechanical Test Capability at - 269°C (4 K)

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ISSN 1754-2979

DOI: https://doi.org/10.47120/npl.MAT102

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Executive summary

This report details requirements for the development of bespoke equipment for characterisation of the mechanical performance of fibre-reinforced polymer (FRP) composite materials at cryogenic temperatures down to -269°C (4 K), the boiling temperature of liquid helium at atmospheric pressure.

The report presents the progress of the first stage of work to specify cryogenic test equipment for FRP composites within the National Measurement System (NMS) project "Polymer Composites Metrology in Support of Regulations, Codes and Standards (RCS) Infrastructure", undertaken by the Advanced Engineering Materials (AEM) Group at the National Physical Laboratory (NPL). The project's key objectives are to develop capability suitable for investigating the mechanical properties at cryogenic temperatures of composite materials used in liquid hydrogen (LH_2) and liquid natural gas (LNG) distribution and storage applications.

The information provided in the report has been collated with inputs from extensive interviews conducted over a period of 9 months with renowned experts within the cryogenics and materials testing fields across Europe and the USA, alongside relevant literature, and existing standards review.

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1 INTRODUCTION

This report details requirements for the development of bespoke equipment for the characterisation of the mechanical performance of fibre-reinforced polymer (FRP) composite materials at cryogenic temperatures with the aim of supporting the liquid hydrogen and liquefied natural gas (LNG) industry towards achieving the Net-Zero.

FRPs are widely used in a significant number of high-performance applications across a variety of industry sectors, including aerospace, automotive, renewable energy and oil and gas. The excellent mechanical properties, low weight, fatigue and corrosion resistance of this class of materials gives them considerable advantages compared to more conventional materials and makes them suitable candidates for use in liquid hydrogen and liquefied natural gas (LNG) storage and distribution applications. As such, the use of FRPs has the potential to reduce fossil fuel reliance, consumption, and greenhouse gas emissions. The application of composites is key to accelerate the transition to a more sustainable and cleaner energy supply, by enabling progress towards a zero-emissions mobility target set by the UK Government. However, full exploitation of composites for these applications is hindered by a lack of understanding of the material performance at cryogenic temperatures, especially for liquid hydrogen applications, as well as the availability of reliable material property data for design.

The development of capabilities for mechanical testing of FRP composites at cryogenic temperatures has been identified as of national strategic importance since it will enable a step change in primarily mechanical characterisation of advanced materials at temperatures as low as 4K. This capability will underpin the development of materials metrology to provide product assurance, increased productivity and competitiveness of advanced manufacturing sectors using composites via clean and sustainable growth.

There are limited sources of literature concerning the characterisation of mechanical performance of FRP composite materials at cryogenic temperatures that present consistent data. It is therefore perceived as of utmost importance to develop test capabilities that can be utilised to develop a validated measurement infrastructure to support the significant increase in demand for reliable and consistent cryogenic materials data that has arisen in recent years.

Due to the emerging growth of industry sectors focussed on the distribution and storage of cryogenic fluids, low temperature test equipment has started to become commercially available. However, at present, sophisticated cryogenic equipment, such as mechanical testing cryostats, are not readily available as off-the-shelf items and require bespoke designs tailored to the use case (e.g., temperature capability, types of tests, load level, etc.).

Due to economic considerations and health and safety concerns [1], the creation of cryogenic test environments almost always utilizes the compression and expansion of gases. At present it is considered appropriate to investigate the performance of FRP composites at cryogenic temperatures (

Table 1), by cooling to the desired temperature using common inert cryogenic fluids such as liquid nitrogen (LN_2) and helium (LHe). It is expected that once the performance of FRPs at cryogenic temperatures becomes increasingly well-characterised, subsequent qualification of materials used for H_2 and LNG applications may be accelerated by proxy testing, as defined in the ATI Fly Zero Advanced Materials report [2]. Specimens could be pre-soaked in liquid hydrogen or LNG and then tested in a non-flammable cryogenic environment, such as liquid nitrogen or helium, to investigate effects linked to chemical interaction.

Table 1. Commonly used cryogenic fluids.

Fluid	Boiling point		Drawbacks
	°C	K	
Liquefied Natural Gas (LNG)	-162	112	Highly flammable and explosive [1]
Liquid Nitrogen (LN_2)	-195.8	77	Considered a safe cryogenic gas with the use of appropriate PPE, oxygen depletion alarms etc, inert and affordable cost (~£0.50 per litre).
Liquid Hydrogen (LH_2)	-252.9	20	Highly flammable and explosive [3].
Liquid Helium (LHe)	-268.9	4.2	Inert cryogenic gas but elevated cost (£20-25 per litre). The use of LHe in large quantities requires significant investment for a Helium recovery system.

2 INDUSTRY DRIVERS

In recent years, attention has focused on the development of new technologies to facilitate the Net-Zero and Carbon Budget Six goals [3], established by the UK Government in 2021. However, there is very limited capability within the UK for measurement of mechanical properties at cryogenic temperatures that satisfies industrial requirements for FRP composite applications that are emerging to ultimately tackle climate change. As a result, many UK organisations have been forced to outsource materials characterisation at cryogenic temperatures to satisfy these requirements to expertise and capability outside of the UK.

The establishment of mechanical testing facilities for characterisation of the performance of FRP materials at cryogenic temperatures, will enable NPL to develop a fit-for-purpose measurement infrastructure and contribute to international standardisation initiatives that will pave the way for wider industrial adoption of composites in sectors such as aerospace and renewable energy. This will form a vital part of the UK's efforts and commitment to reach net-zero, as well as positioning the UK to become a global leader in low-carbon hydrogen technologies and to secure future economic and job opportunities.

According to the UK Hydrogen Strategy report published in August 2021 [3], the use of hydrogen and LNG, amongst other decarbonised fuels, will play key roles in improving energy efficiency and reducing CO_2 emissions.

The Aerospace Technology Institute's (ATI) FlyZero project has identified liquid hydrogen as a green fuel with the potential to become the principal candidate for zero-carbon emission combustibles in the future of commercial aviation [2], [4]. Amongst the benefits of liquid hydrogen, it should be highlighted that its combustion could reduce the impact on climate change by commercial flight by 50-75%, whereas used in fuel-cell propulsion this change is 75-90% [4]. These targets are based on the fact that CO_2 emissions can be eliminated when liquid hydrogen is burnt as a fuel and that hydrogen fuel cells can provide 10 times the energy of lithium-ion batteries. In the UK, some major aerospace organisations are already developing solutions to liquid hydrogen powered commercial flights and other areas of transport in their transition to net-zero [2].

The key role of LNG in the global energy market relies on the significant environmental benefits that can be achieved by replacing coal with LNG. To tackle climate change, LNG is an excellent candidate to reduce greenhouse gas emissions due to its high energy density and low CO_2 release.

To date, the use of FRP composites has already played a significant role in reducing fossil fuel consumption and greenhouse gas emissions by enabling lighter weight vehicles (e.g. aircraft, cars etc) and renewable energy applications (e.g. wind turbines). A greater uptake of FRP composites will be required to enable efficient hydrogen and LNG storage and distribution applications. This will lead to significant technology development in the UK as well as the generation of economic prosperity as progress is made toward achieving net-zero by 2050.

3 MECHANICAL TESTING OF FIBRE-REINFORCED POLYMER (FRP) MATRIX COMPOSITES AT CRYOGENIC TEMPERATURES

As a result of extensive research and development conducted since the 1970s to support the insulation of superconducting magnets [5, 6], alongside the evolution of FRP composites for spacecraft and launch vehicle applications since the first Apollo moon landing in 1969, the need to understand the effects of cryogenic temperatures on FRP composite materials has been essential. However, it is only recently that advanced composites have been identified and subsequently developed, as candidate materials for hydrogen and LNG storage applications.

Accurate measurement of the mechanical performance of FRP composites over a range of cryogenic temperatures (down to as low as ~4K) is essential in order to generate confidence in their use for cryogenic applications. The availability of material property data at cryogenic temperatures will enable and validate designs, as well as ensuring the quality of finished components. It is well-known that the mechanical properties of FRP composites undergo noticeable changes as a function of temperature [6, 7, 8]. Wu et al. [9] state that cryogenic temperatures have a significant effect on the polymeric matrix, with the composite becoming brittle due to reduction in polymer chain mobility. At cryogenic temperatures, increases in strength, modulus, and fracture toughness of FRP composites have been observed compared to room temperature properties [6, 7, 9, 10]. Researchers have also noted the generation of microcracking damage when cycling between ambient and cryogenic temperatures without the application of mechanical loading, owing to the thermal expansion mismatch between the matrix and fibre [2, 9, 10]. Hence, the extensive characterisation of mechanical properties under these conditions is of paramount importance to determine material performance and assurance.

However, at present there is little evidence of studies describing in detail the specifications of experimental apparatus required or addressing the challenges to successfully undertaking mechanical characterisation at extremely low temperatures. In addition, it should be highlighted that several authors have reported inconsistency and significant uncertainty in mechanical properties of FRP composites measured at cryogenic temperatures [6, 7, 11].

Therefore, one of the main constraints that arises when testing FRP at cryogenic temperatures is not only the limited number of public sources covering test procedures and equipment, but perhaps most importantly the lack of standard test methods. The lack of a fit-for-purpose measurement infrastructure (e.g., equipment, jigs, gripping systems, standards etc.) may lead to designers overlooking the benefits of composites and instead default to the use of metals due to the increased likelihood of data availability. Test standards covering methods for the measurement of properties at extreme conditions, such as cryogenic temperatures, are essential to meet safety regulations and provide product assurance. New test standards providing guidance on mechanical testing of FRP composites at cryogenic temperatures, including details for apparatus for such tests, will need to be developed quickly to meet industrial requirements, particularly those of the commercial aerospace and energy storage/distribution sectors who are looking at the use of FRP composites now.

Unlike FRPs composites, other type of materials, such as metallic alloys, have been extensively used for cryogenic applications for many decades, and the associated research has been widely reported and standardised.

Therefore, a brief review of existing standard test methods developed for characterising the behaviour of metallic specimens at cryogenic temperatures has been undertaken:

- **JIS Z 2277:2000** Method of tensile testing for metallic materials in liquid helium [12].

The method defined in JIS Z 2277:2000 focuses on the measurement of proof stress, tensile strength, elongation after fracture and reduction of area of metallic materials by applying lengthwise tensile forces to the specimen submerged in liquid helium under atmospheric pressure. Tensile tests undertaken in accordance with this Japanese Standard, require a displacement control-type tensile test machine equipped with both a cryogenic device and an extensometer.

Additional requirements concerning the cryogenic apparatus defined in the standard are listed below:

- The cryogenic apparatus, or cryostat, should be composed of a dewar capable of gripping and loading a test piece submerged in liquid helium.
- The dewar should have a heat insulating layer with sufficient capacity to maintain the temperature of a test piece at ~4 K.
- Gripping devices should have suitable strength, rigidity, and low brittleness for testing at 4 K; the use of austenitic steels or titanium alloys are advised.
- The cryogenic apparatus should ensure alignment whilst running tensile tests. An inspection method for eccentric load of cryogenic apparatus is suggested to ensure bending does not exceed 10% whilst running tests.
- The cryogenic apparatus should be equipped with level gauges for ensuring specimens are fully submerged in LHe thorough the test. Measuring the temperature of the specimen during the test it is not required.
- An extensometer suited for cryogenic temperatures should be calibrated and verified before use. Guidance on the calibration of extensometer for cryogenic tensile tests is also provided within the standard.

- **BS ISO 19819:2004** Metallic materials —Tensile testing in liquid helium [13] This British Standard was withdrawn in October 2015 and replaced by BS ISO 6892-4:2015. This was the first ISO standard test method released to provide guidance for the testing of metallic materials al cryogenic temperatures.

- **BS EN ISO 6892-3:2015** Metallic materials — Tensile testing — Part 3: Method of test at low temperature [14]. The third part of ISO 6892 focuses on tensile testing of metallic materials at low temperatures, between +10°C and -196°C.

  The method consists of straining a test piece under the application of tensile load at low temperature for the determination of mechanical properties. In this standard, two test speed regimes are proposed; (i) the first regime focuses on minimising the variation of test rate (with tolerances of ±20 %) based on strain rate control when measuring strain rate sensitive properties and estimating measurement uncertainty, (Method A), and (ii) the second regime is based upon expanded strain rate ranges and tolerances (Method B).

  According to ISO 6892-3, specimens should be cooled to the specified temperature and soaked for 10 minutes before loading. In addition, different methods for cooling are suggested:

  - by a refrigeration unit,
  - by expansion of compressed gas (e.g., CO_2 or N_2),

- by immersion in a liquid maintained at its boiling point (e.g., N_2) or in a refrigerated liquid.

It is also acknowledged that the type of cooling plays a significant role on the cooling time (i.e., time to reach the desired test temperature) and on the heat transfer during the test, and as a result might have a significant influence on the test result.

This part of ISO 6892 provides generic advice concerning instrumentation, gripping and measurement of temperature. The temperature measuring equipment should be calibrated and verified over the working range once per year, and should have a resolution equal to or better than 1 °C and an accuracy of ±2 °C for the range +10 °C to -40 °C and ±3 °C for the range -41 °C to -196 °C. For recommendations on gripping of test samples, the standard refers to ISO 6892-1: Method of test at room temperature [15], where general advice on gripping at room temperatures is provided.

- **BS ISO 6892-4:2015** Metallic materials — Tensile testing Part 4: Method of test in liquid helium [16]. This International Standard provides guidance on tensile testing of metals immersed in liquid Helium at –269 °C (4 K). It supersedes BS ISO 19819:2004 [13].

ISO 6892-4 describes the method for tensile testing of metals at cryogenic temperatures (less than –196 °C), which requires special apparatus (a cryostat), instrumentation and sub-size test pieces. This standard also addresses serrated yielding, adiabatic heating, and strain-rate effects associated with metallic samples tested at cryogenic temperatures.

To conduct a tensile test according to this part of ISO 6892, specimens are fully immersed in LHe in a mechanical cryostat (Figure 1), and then tested in displacement control at a nominal strain rate of 10^{-3} s^{-1} or less. General requirements regarding tensile cryostats, support apparatus and instrumentation are described. The key features of a tensile cryostat in accordance with ISO 6892-4:2015 [16] are as follows:

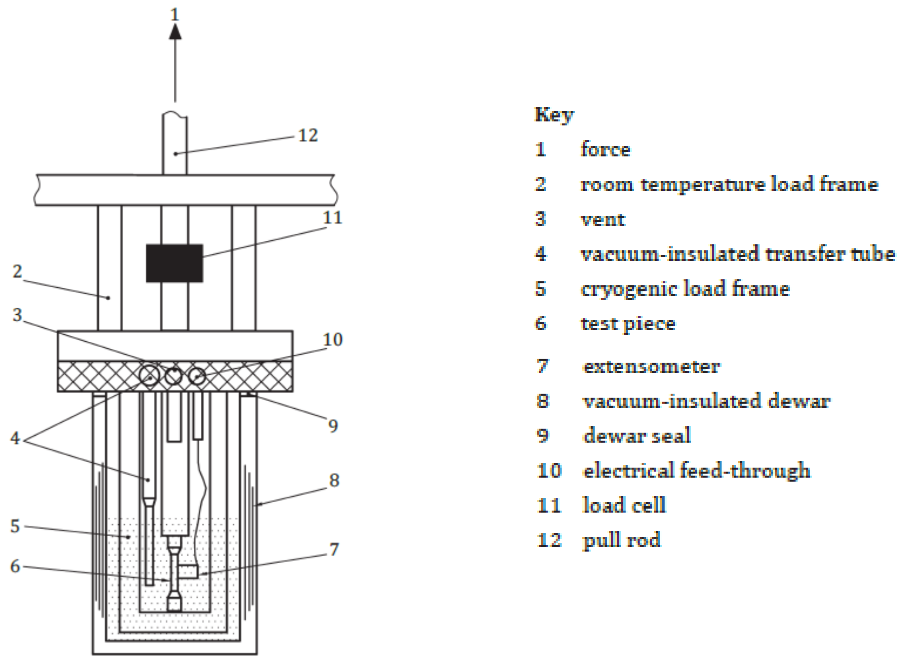

Figure 1. Schematic illustration of cryostat for tensile testing at 4 K. Source: BS ISO 6892-4:2015 [16]

It should be noted that this standard emphasises the importance of alignment to minimise bending strains in tensile tests. The maximum acceptable bending strain should be Class 10 in accordance with ISO 23788 [17]. Several options are proposed to calculate the maximum bending strains with respect to different specimen geometries.

ISO 6892-4 states that metallic alloys tested at cryogenic temperatures can often double or triple the measured room temperature strengths. Hence it is recommended to use sub-sample sizes as an alternative when standard test pieces are inappropriate to ensure specimens can be tested in conventional test machines of 100 kN or less. The use of sub-size test pieces is designed to minimise the thermal mass of coupon, cryostat and jigs used for loading, thereby increasing the thermal efficiency of the cryostat and reducing the cooling time. Note that for testing of FRPs, the use of sub-size coupons is not possible due to the architecture of material formats, lay-up and the requirement to encompass a representative volume of material or number of unit cells for a woven material.

The cooling procedure defined requires the tensile cryostat to be pre-cooled using liquid nitrogen. Once thermal equilibrium at the boiling point of nitrogen is achieved (–196°C, 77 K), all the nitrogen should be removed from the cryostat, and LHe should be transferred into the cryostat until the specimen and grips are fully immersed. Specimens should be maintained at 4 K thorough the duration of the tests.

It is also highlighted that special attention should be taken to consider the formation of condensation when testing at cryogenic temperatures. Ice can block transfer lines; and therefore, it is key to prevent icing by drying the inner part of the cryostat thoroughly with an air jet.

- **ASTM E1450 – 16** Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium [18]. This standard describes a procedure for conducting tensile testing of structural alloys by immersing specimens in liquid helium in a tensile cryostat.

  The standard includes modifications for cryogenic testing which requires special apparatus, instrumentation, and smaller specimens. This standard also covers concerns for serrated yielding, adiabatic heating, and strain-rate effects associated to metallic samples tested at cryogenic temperatures.

  To conduct a tensile test in accordance with this standard, the specimen is loaded into a tensile cryostat, fully immersed in LHe and tested in displacement control at a nominal strain rate of 10^{-3} s^{-1} or less. Tests using force control or high strain rates are not considered.

  The stress-strain response of a material tested in liquid helium depends on whether force control or displacement control is used. Crosshead displacement control is specified in this standard.

  As a recommendation concerning apparatus, the standard suggests measuring the compliance of the cryogenic test frame by coupling the force train with a rigid block or calibration specimen and then measuring the compliance at a low force and at the highest force expected in use.

  The heat-transfer characteristics of gaseous helium are inferior to those of liquid helium therefore, according to this standard it is imperative that the specimen remains submerged in liquid helium to minimize the influence of generated heat on the mechanical property measurements.

As previously described in [16], sub-size specimens are also recommended in this standard, since conventional 100 kN test machines may be insufficient for testing full-size specimens. Sample preparation steps, such as machining, surface finish, and alignment can have a greater influence on properties as the specimen size is reduced.

Generic guidance concerning tensile cryostats, alignment, gripping mechanisms and measuring devices is provided in this standard. In addition, advice regarding construction materials that have proved successful for design of grips, pulls rods and tensile cryostat frames is provided.

- **ISO 21028-1:2016** Cryogenic vessels — Toughness requirements for materials at cryogenic temperature — Part 1: Temperatures below -80 degrees C. [19]. Part 1 of standard 21028 defines the toughness requirements of metallic materials used in cryogenic vessels.

Once the development of suitable standards reaches maturity, it is expected that long-term extensive cryogenic qualification campaigns will be required to predict knock-down factors used in design and ultimately support the definition of material allowables to meet hydrogen and LNG storage requirements [2]. In addition, ATI foresees that in the long-term small testing campaigns in a representative environment, such as hydrogen, might be required to confirm initial insights based upon pre-exposed and conventionally tested materials, as defined in Section 1.

4 MECHANICAL TEST SYSTEM

The Mechanical Test Facility (MTF) at NPL offers a wide range of quasi-static and dynamic state-of-the-art test machines to support the characterisation of advanced materials for all sectors of industry. The addition of a dedicated cryogenic mechanical test machine will provide the capability for underpinning research to be undertaken to support the characterisation of polymer matrix composites at temperatures as low as 4 K.

The cryogenic mechanical test system will consist of a servo-hydraulic test machine upon which will be mounted a mechanical cryostat in which test samples can be immersed in various cryogenic liquids/gases depending on the desired test temperature. A servo-hydraulic test frame (Figure 2) is required to perform quasi-static and dynamic fatigue tests. In addition, the hydraulic actuator on the test machine will be mounted at the top of the frame to facilitate mounting of the cryostat on a T-slotted baseplate, thereby enabling easier separation of the cryostat components upon specimen retrieval and replacement. After careful consideration of the anticipated load levels required to fail specimens of different material types, formats and specimen dimensions, a 250 kN load cell has been specified as suitable to fulfil current and future FRP composite testing requirements. As such, a 250 kN rated test frame is therefore required, as well as a cryostat of equivalent rating. It is important to note that when testing specimens, it is the columns within the cryostat itself that react the applied load and not the columns of the mechanical test machine. An example of a suitable hydraulic test machine with top mounted actuator is shown in Figure 2.

The principal features required for a cryogenic mechanical test system suitable for testing of FRP composites, as well as other classes of material, are as follows:

- A servo-hydraulic test machine with a load frame featuring two extended columns to ensure robustness.
- Telescopic lift cylinders that allow raising and lowering the top part of the cryostat.
- Top-head actuator in crosshead, 250kN loadcell load cell on actuator.

- Actuator displacement control and measurement with an accuracy of ± 0.2% approximately.
- The testing machine, load cell and instrumentation must be verified and calibrated in accordance with ISO 7500-1 or per supplier requirements.
- Extra height frame to provide sufficient daylight between base and crosshead.
- T-slotted platen to securely mount and clamped down the cryostat.
- Data acquisition software compatible with temperature controller.
- At least 4 channels for transducers.
- Fixturing options (Tension grips, compression, and fatigue test fixtures) for different sizes and specimens' shapes.
- Instrumentation suited for cryogenic temperatures. Reliable and accurate extensometers to measure strains in the gauge length of full-size specimens during loading at 4 K are required. Alternatively, strain gauges well suited for cryogenic testing of CFRP could be used when load at failure is desired.

Figure 2: Example of a servo hydraulic test machine with top mounted actuator. Source: Instron.

5 MATERIALS TEST CRYOSTAT

To characterise mechanical properties at cryogenic temperatures, it is essential to make use of specialised equipment capable of controlling accurately displacement, load, strain rate, and desired temperature between room to cryogenic temperatures, down to -269 °C (4 K).

As defined in ASTM E1450-16 [18], to conduct a tension test at cryogenic temperatures, the specimen is submerged in liquid helium in a tensile cryostat. A tensile cryostat, shown in Figure 4, is described in the same standard as “a test apparatus for applying tensile forces to test specimens in cryogenic environments”.

Cryostats for mechanical testing of materials are complex and fully tailored equipment that are generally not procured off-the-shelf. Cryostats consist of two main components, a load train and a cryogenic dewar [20]. Based upon extensive investigations [21, 8, 22, 20] and advice provided by experts on the subject matter, NPL considers that the basic thermal, structural, instrumentation and H&S requirements for a cryostat suited for mechanical testing of FRP composites should be as follows:

Figure 2. Cryogenic Testing Station. Source: MTS

5.1 STRUCTURAL

It should be highlighted that mechanical properties of FRP composites are highly sensitive and influenced by temperature and loading mode, hence special attention should be paid to parameters such as alignment, test control (displacement, load, or strain), and uniformity of the temperature in the cryostat and along the specimen length [21].

The cryostat must be designed to ensure mechanical stability whilst being fully integrated with the servo-hydraulic fatigue test system, mounted between the T-slotted base and the underside of the upper crosshead in order to minimize heat losses while maintaining very low temperatures, and alignment with the machine actuator axis during the tests. The load frame of the cryostat system should act in compression to react the specimen’s tensile stress;

consequently, the load should be reversed to perform compression tests. The cryostat system should be modular and easily converted for static and dynamic tests.

As shown in Figure 4, a cryostat for mechanical testing should comprise of internal posts (E), outer rods (F), a top flange (C), inner and outer bottom plates (D) and bottom flange (G). The system should employ adjustable force-columns to facilitate alignment. The transmission of the load should be conducted by means of 3 or 4 internal posts used in compression to react specimen tensile force [21, 8].

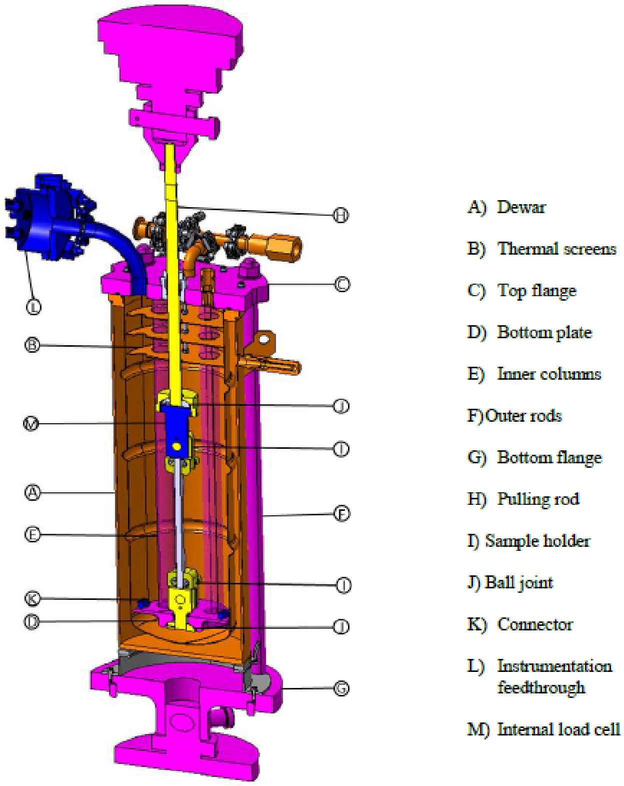

Figure 4. Schematic example of a tensile cryostat showing the main features of the system. Source: [21]

The cryostat should feature sets of pull and push rods (Figure 4, H) which should be easily interchanged in order to achieve tensile or compressive loading and gripping (e.g., tension, compression, fatigue). Pull rods or specimen bearing members should carry the load and be capable of performing at extreme low temperature. The bottom end of the load train should be built with a clevis pin design, to ensure central alignment of the specimen with the load axis.

Sample holders and compression platens should also be considered in the design of the cryostat, and sufficient space within the dewar should be designed in to fit grips and fittings as well as enabling sample alignment. According to Aviles Santillana et al. [21] shoulder-headed sample holders (Figure 4 4, I) minimise stress concentrations in tensile specimens. However, it is expected that samples holders would need redesign to cater for FRP coupons.

Cryogenic materials with low thermal conductivity and low embrittlement, such as titanium base alloys (Ti-6Al-4V), austenitic stainless steel (AISI 316L or AISI 304LN), maraging steels (200, 250, or 300 grades, with nickel plating to prevent rust) [18], and Inconel 718 [21, 23], alongside resistive heating elements must be used in the construction of the cryostat to avoid failure in service and optimise cryogenic thermal efficiency.

The outer dimensions of the cryostat should be limited to the space between the servo-hydraulic test system columns and should be easily mounted and dismounted. The test space within the dewar must be sufficient to fit full-size specimens in accordance with relevant standard geometries. Typical FRP specimen sizes are shown in Table 2:

Table 2. Commonly used standard test methods of fibre-reinforced plastic composites.

Standard number	Specimen dimensions		
	Length (mm)	**Width (mm)**	**Thickness (mm)**
BS EN ISO 527-4:2021 Plastics — Determination of tensile properties	≥ 250	25 ± 0.5 or 50 ± 0.5	2 to 10
BS EN ISO 527-5:2009 Plastics — Determination of tensile properties	≥ 250	15 ± 0.5 or 25 ± 0.5	1 ± 0.2 or 2 ± 0.2
ASTM D3039/D3039M – 17 Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials	≥ 250	15 ± 0.5 or 25 ± 0.5	1 ± 0.2 or 2 ± 0.2
BS EN ISO 14126:1999 Fibre-reinforced plastic composites — Determination of compressive properties in the in-plane direction	110 ± 1	10 ± 1	2 ± 0.2 or 10 ± 0.2

5.2 THERMAL

Heat losses are caused by three main mechanisms: conduction, convection and radiation [8]. Therefore, one of the main challenges for the design and manufacture of a cryostat system for mechanical testing is optimising thermal efficiency, due to the dimensions and thermal mass of the system. The volume of the cryostat test dewar for mechanical testing is approximately 20-25 litres. As a result, the approach suggested by [21] is minimising the mass and increasing the contact surface area with the coolant, which should also avoid heat losses in order to minimise cryogen consumption and increase cooling speed.

The most important element of a cryostat in terms of thermal performance is the chamber or dewar (Figure 4, A), which should consist of a double-wall vessel in which an outer-jacketed dewar of liquid nitrogen surrounds the inner dewar of liquid helium. According to Aviles Santillana et al. [21] and ISO 6892-4:2015 [16], these type of dewars have proved a more efficient solution, although of great complexity, in terms of time efficiency and cryogen consumption.

The dewar should feature inlet and outlet ports in the top flange (Figure 4, C), a suitable cryogen dispenser mechanism and sensors to control and monitor temperature within the chamber and on the specimen. Liquid-level indicators, flow valves and meters for cryogen flow control are required to monitor that the specimen and chamber remains at the desired temperature thorough the test. In addition, vacuum pumps, and at least two exhaust ports are required, one for each pressurized gas dewar (LN_2 and LHe). Thermal screens or baffles should be used in the top part of the cryostat to decrease heat exchange with the top plate of the cryostat and o-ring seals should prevent the gas to scape (Figure 44, B) [21].

The cryostat should be able to achieve any fixed-point temperature in the range from ambient to 4 K with temperature control accuracy of ±2 K. Cooling is provided by supply of liquid or gaseous nitrogen and/or helium, from vacuum insulated dewars and transfer lines. The cryogenic temperatures of -196°C (77 K) and -269°C (4 K), boiling points of liquid nitrogen and liquid helium, respectively, should be obtained by filling the dewar and immersing the test specimen and fixture. In addition, to allow for versatility in terms of temperature range, the cryostat should be capable of conducting temperature-controlled tests at intermediate test temperatures in the range from 20 K to 300 K. The cryostat and test fixturing should be instrumented with temperature sensors and resistive heating elements. LN_2 is used as the cooling medium over the range of temperatures from room to -196°C (77 K), whereas any tests carried out at temperatures below this point, down to -269°C (4 K), will additionally require liquid helium, as shown in Table 3.

It is expected that a ~20-25 litre cryostat for mechanical testing would require between 25-30 litres of LHe for 30 minutes to cool down a specimen when optimal thermal conditions are achieved [21]. After testing, nitrogen and helium may be released to the atmosphere. However, helium can be recycled as a gas, and then reliquefied, due to its high price.

The cryogenic thermal design is a key part of the whole design of a cryostat for mechanical testing, but it must never compromise mechanical stability and/or safety.

Table 3. Overview of temperature ranges, cooling methods and equipment. [23]

Cryogenic temperatures of interest		Cooling methods	Equipment
K	°C		
112	-162	Evaporative cooling of liquid nitrogen	Nitrogen dewar – gas phase
77	-195.8	Direct immersion in liquid nitrogen	Nitrogen dewar – liquid phase
20	-252.9	Direct immersion in liquid nitrogen and evaporative cooling of liquid helium	Helium dewar – gas phase
4.2	-268.9	Direct immersion in liquid helium	Helium dewar – liquid phase

5.3 INSTRUMENTATION

As pointed out by Aviles Santillana et al. [21] when designing a cryostat system for mechanical testing, attention should be paid to ensure that sufficient sensors are in place for accurate monitoring of displacement, load, strain, and temperature to be achieved. Electrical connectors (Figure 4, K) for the instrumentation feedthrough (Figure 4, L) are required.

Clip-on axial and/or biaxial extensometers, linear variable differential transformer (LVDT) displacement transducers and crack opening displacement (COD) gauges rated for cryogenic temperatures to account for different specimen sizes and tests are required. If strain to failure is desired, strain gauges suited for cryogenic temperatures can also be used. At present, these strain measurement devices are all commercially available from well-stablished instrument manufacturers. Supplier calibration and user verification at the desired cryogenic temperatures is essential to ensure data accuracy and is recommended to be of Class B1 or better as per ASTM E 83 [24].

5.4 HEALTH AND SAFETY

The highest priority for the adoption of FRP composites for use in the hydrogen and LNG storage sectors is safety. Due to safety concerns [1], the characterisation of FRP composites at cryogenic temperatures, will be mainly performed by cooling down using common inert cryogenic fluids, such as liquid nitrogen (LN_2) and helium (LHe).

ASTM E1450 [18] addresses safety concerns associated with the use of these cryogens for mechanical testing purposes. The main safety risks associated with using LN_2 and LHe are burns and asphyxiation due to oxygen depletion. All users who work with cryogenic gases or systems using such gases (e.g., in dewars or environmental chambers), should undergo specific training prior to starting to work with cryogenics. Theoretical and practical training must be carried out to become thoroughly familiar with the properties, safety considerations, first aid measures and main hazards of the use of cryogenics.

Several precautions must be taken when using cryogenic fluids and equipment using such gases. Even for applications where there should not be direct interaction with cryogenic liquids as such, Risk Assessments must be in place and appropriate personal protective equipment (PPE) must be worn at all times. Users should wear safety goggles, safety boots, lab coat, face shields, cryogenic gloves for handling cold specimens or instruments, as well as portable oxygen depletion monitors. In addition, areas where cryogenic liquids are to be used, must be of suitable size, well ventilated and wall mounted oxygen depletion alarms must be operational.

Safety alarms and pressure relief safety valves (<0.5 bar) welded into the feedthrough spool should be fully incorporated to the control system of the cryostat to ensure the operation of the equipment is safe at all times, whilst running of the machine.

6 MEASUREMENT CHALLENGES AND BARRIERS TO TECHNOLOGY ADOPTION

Previous sections of this report detail the background and requirements to develop capabilities for measuring mechanical properties of FRP composite materials at cryogenic temperatures. Technical challenges expected in developing such capabilities for cryogenic testing that must be addressed are summarised below:

- Lack of standard test methods — The lack of international standards for measuring mechanical properties of FRP composites at cryogenic temperatures presents a significant drawback for the adoption of composites compared to metals. It is believed that the significant scatter across literature generated at cryogenic temperatures is caused by the lack of standard test methods, apparatus, and instrumentation for measuring mechanical properties under these extreme conditions.

- Specimen sizes — Unlike with metallic specimens [18], FRP specimen sizes cannot be reduced. Sub-size specimens are not considered suitable when testing composites since specimen dimensions are standardised to avoid boundary conditions, such as free-edge effects, and to account for material variability representative volume of material and layup.

- Temperature maintenance — Vacuum insulated dewars or transfer lines capable of retaining liquid nitrogen and helium are required to achieve and maintain cryogenic temperatures thorough the test. After testing, helium may be released to the atmosphere, recycled as a gas, or reliquefied. Reliquefication of helium requires additional capabilities in purification and support systems.

- Cooling efficiency — The cooling method, as well as the cooling rate, should also be standardised to generate consistent results. As stated in [16] the type of cooling medium, whether specimens are cooled down by refrigeration, LN_2 gas or immersion, has a significant effect on the heat transfer and therefore, the results. Significant efforts would be needed on increasing the rate at which mechanical tests can be carried out.

- Alignment — Robust system alignment is essential to avoid bending strains that might affect the results. Limited published sources [13, 16, 17, 18, 21] address guidance to ensure alignment when designing and using cryostats for mechanical testing.

- Gripping mechanisms — Different types of gripping need to be considered to ensure proper load distribution is applied to specimens and avoid stress concentrations at free-edge effects when clamping. Some gripping approaches are proposed in existing literature [7, 21], such as shoulder-headed, threaded, wedged, or pinned connections, however further investigations are required in order to find the optimal gripping methods for FRP composites at cryogenic temperatures.

- Instrumentation — Guidance and standardisation on instrumentation requirements to achieve the required response frequency for adequate mechanical characterisation when testing composites at cryogenic temperatures is essential. The use of bonded strain gages at cryogenic temperatures, however, requires precautions not required at room temperature. There are two major constrains: the gauge factor varies with temperature, and thermal output is introduced as the specimen-gauge combination is cooled from room temperature to 4 K. For this reason, it is believed that strain gages generate less accuracy than extensometers, when testing at cryogenic temperatures, as reported in literature [6, 7].

- Measurement uncertainty — The complexity of cryogenic testing in comparison to standard room temperature testing places an additional challenge on ensuring data quality and an understanding of measurement uncertainty. ISO 6892-3:2015 [14] estimates the uncertainty associated with testing at low temperatures as the reproduction of standard ambient temperature uncertainty with the addition of temperature, type of cooling medium and strain rate components. The variations in temperature and the type of cooling medium have been found to have a noticeable potential effect on testing results.

In addition to technical challenges, it is expected that barriers to technology adoption [25] for measuring mechanical properties at cryogenic temperatures, that would allow greater incorporation of composites in the hydrogen and LNG sectors, will include:

- Technological uncertainty: The main need to develop capabilities for measuring mechanical properties of FRP composites at cryogenic temperatures, is driven by hydrogen technology. Further R&D programs and funding are required to continue developing cryogenic technology to further enable the use of FRP composites before they can be widely deployed in the hydrogen and LNG storage applications towards zero carbon emissions. Therefore, the application of composites along with the development of technologies and infrastructure for hydrogen will need to be proven at scale before they can be widely deployed [3].

- Initial investment Unique skills and expertise are key to master the design, manufacture and use of cryostats for mechanical testing of materials. Cryogenic experience, as well as a profound knowledge of mechanics of materials are two specialised fields of expertise and their study requires access to capital equipment suited for the application which involves significant cost. In addition, other recurring costs, such as the consumption of large volumes of LN_2 and LHe need to be considered.

- Lack of expertise Skills, know-how and good practice related to mechanical testing of materials at cryogenic temperatures are required. Adequate theoretical and practical knowledge, as well as cryogenic experience are essential to assess the behaviour of materials at cryogenic temperatures.

- Limited sources of materials data Little published evidence of reliable materials data at cryogenic temperatures are required to validate the performance of FRP composites in hydrogen and LNG storage applications [25].

7 CONCLUSIONS AND FURTHER WORK

A fuller understanding of the relationship between the material properties of FRP composites at ambient and cryogenic temperatures will allow optimised structural design and provide assurance to meet the requirements of emerging advanced manufacturing sectors.

The development of capabilities for mechanical testing of FRP composites at cryogenic temperatures is considered vital to support the demand for key technology growth in sustainable sector, such as hydrogen and LNG storage, that has arisen in the recent years.

In addition, a key recommendation from this work is to accelerate the standardisation and publication of technical documentation to provide guidance on testing methods, specific apparatus, and instrumentation for measuring mechanical properties of FRP composites at cryogenic temperatures.

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