National Physical Laboratory

AEROSPACE TECHNOLOGY INSTITUTE

Hydrogen Capability Network

International landscape on cryogenic and hydrogen materials testing

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Funding acknowledgement

This work was funded by the UK Government's Department for Science, Innovation & Technology through the UK's National Measurement System programme, the International Science Partnerships Fund (ISPF non-ODA), and the Department of Business and Trade (DBT) through the ATI's Hydrogen Capability Network.

Stakeholder acknowledgement

NPL would like to thank all stakeholders listed in the Appendix - Key Contacts who provided input into this report either directly or by participating in discussions during the laboratory visits.

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Executive summary

Extensive work and stakeholder engagement by the National Physical Laboratory and the Hydrogen Capability Network (HCN) at the Aerospace Technology Institute identified a clear lack of UK-based test infrastructure both for material level characterisation as well as system level testing, relevant to cryogenic and liquid hydrogen technology development. It has also been identified that test infrastructure and expertise exist in Europe and America and as such, NPL and HCN teams have identified, engaged, and organised several visits aiming to host joint dialogues with organisations that have expert knowledge on materials testing at cryogenic conditions and handling of liquid hydrogen; and determine the infrastructure capability, capacity, and programmes of work needed to develop competence in support of UK supply chains with emerging energy and environment technologies.

The key takeaways of the fact-finding mission to the European and American laboratories are summarized in this report. It is vital that the UK develops measurement infrastructure to underpin hydrogen technology development by UK-based organisations and support innovative technology supply chains. The infrastructure should be inclusive of cryogenic and in-situ liquid hydrogen testing as well as in-situ gaseous hydrogen testing. More specifically:

- For cryogenic testing, there is an immediate need for both capability and capacity to cover the increasing demand for evaluation of mechanical and thermal properties of materials at 20 K.
- For in-situ liquid hydrogen testing, there is an immediate need for research-based testing facilities i.e., utilising small-scale hydrogen liquification including in-situ, to boost materials research and innovation, followed by investment in large-scale facilities like those at NASA Marshall Space Flight Center (MSFC).
- For in-situ gaseous hydrogen testing, the coordination of existing facilities must be prioritised together with an increase of current capability to higher pressures and temperatures similar to those at Sandia Laboratories.

Authors

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Stefanos Giannis is the Science Lead in the Advanced Engineering Materials Group at NPL, setting the strategic direction for the development of key materials metrology infrastructure - across polymer composites, advanced alloys and AM materials and engineered surfaces & coatings - to support and enhance the advanced materials regulatory infrastructure. He has 20+ years postgraduate experience in polymers and polymer composites testing, inspection, and certification. He is a Fellow of the Institute of Materials Minerals and Mining (FIMMM) and Visiting Professor at the School of Mechanical Engineering Science in the University of Surrey.

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Mr Mike Gower is a Principal Scientist within the Advanced Engineering Materials group at the National Physical Laboratory. Mike is responsible for the development of metrology to underpin the use of composites as multifunctional materials to enable optimisation of the processing and in-service performance of composite structures. He has 29 years of experience in mechanical testing, physical analysis, characterisation, non-destructive testing, and finite element analysis of composite materials. Mike holds a degree in Aeronautical Engineering and an MSc in Composite Materials from Imperial College.

Nassos Spetsieris

Nassos Spetsieris is a Higher Scientist within the Advanced Engineering Materials group, responsible for developing the capability of cryogenic mechanical testing of advanced materials, in support of adopting hydrogen as a sustainable fuel. He has recently co-authored a report, exploring the challenges around cryogenic storage from a materials and standards perspective, as well as presented and chaired in international cryogenics conferences.

Petra Mildeova

Petra Mildeova is a Senior Scientist within the Advanced Engineering Materials group at the National Physical Laboratory. She leads the thermal properties testing of advanced materials and is currently focussed on developing cryogenic capability in this area. Petra has more than 10 years' experience in materials characterisation and analysis and thermal properties testing.

Huw Edwards

Huw Edwards is the Technical Lead for the Hydrogen Capability Network (HCN) within the Aerospace Technology Institute (ATI). He is responsible for the technical strategy within the HCN and is working to identify and address the key gaps to enable Liquid Hydrogen commercial aircraft. He has been in the aerospace sector for over 10 years, with the last 5 being focused on the development of liquid hydrogen technologies for commercial aviation. His initial interest in this area formed through the development of composite liquid hydrogen tanks and associated cryogenic material characterisation techniques for which he is currently in the final stages of an EngD.

Contents

Introduction

The aviation industry emitted 915 million tonnes of CO_2 in 2019, roughly 2% of total global CO_2 emissions [1]. At current rates and without mitigation, aviation is expected to become one of the largest emitting sectors by 2050, therefore, decarbonising current operational practices is critical to achieving net zero. A key route in achieving this is via transitioning to alternative fuels and several industry players, backed by government funding, are investigating the transition to liquid hydrogen.

The Aerospace Technology Institute's (ATI) FlyZero project set out a vision for the future of zero-carbon emission flight and presented concepts for the next generation of aircraft powered by liquid hydrogen. According to the FlyZero project's findings green liquid hydrogen (hydrogen produced by splitting water into hydrogen and oxygen using renewable electricity) is the most viable pathway to decarbonisation when compared to other zero-carbon emission energy sources such as batteries and ammonia and, could power large aircraft utilising fuel cell, gas turbine and hybrid propulsion systems. The project identified six technologies fundamental to hydrogen fuel cell or hydrogen gas turbine aircraft [2], these being:

1) Hydrogen Gas Turbines & Thrust Generation
2) Fuel Cells
3) Cryogenic Hydrogen Fuel System & Storage
4) Thermal Management
5) Electrical Propulsion Systems, and
6) Aerodynamic Structures.

A key underpinning cross cutting technology brick is that of Advanced Materials, for which FlyZero recommended the establishment of new, UK-based, hydrogen materials test facilities and dedicated funding for research and development of materials used in hydrogen environments [3]. As a direct consequence, the Hydrogen Capability Network (HCN) was established at the ATI tasked with defining an operating model for a group of open-access facilities designed to accelerate the development of liquid hydrogen (LH_2) aircraft technologies and capabilities.

A globally leading aircraft engine manufacturer, Rolls-Royce (RR), have also announced programmes to demonstrate aircraft engines can be operated safely using liquid hydrogen [4], however, materials used in future components will need to be certified for safe operation. In parallel, Airbus have communicated their ambition to bring to market the world's first hydrogen-powered commercial aircraft by 2035 [5], exploring a variety of configurations and technologies through the ZEROe project, as well as preparing the ecosystem that will produce and supply the hydrogen. In all cases, qualification of materials will rely on the availability of infrastructure that can provide validated and traceable test methods and measurement standards, for the determination of material properties to support these emerging technologies.

Separately, NPL has been approached by several organisations for provision of materials data that will support cryogenic hydrogen technology development. As a result, in partnership with other UK organisations, NPL a workshop to identify challenges in realising liquid hydrogen cryogenic storage from a materials and standards perspective, as well as highlighting priority data needs [6]. This work also identified the requirements for developing measurement methods and standards to support industry sectors aspiring to develop cryogenic hydrogen technologies.

A common theme apparent amongst stakeholder engagement was the lack of UK-based test infrastructure both for material level characterisation as well as system level testing. It has also been identified that test infrastructure and expertise exist in Europe and the USA and as such, NPL and HCN teams have identified, engaged, and organised several visits to organisations in Europe and the US aiming to:

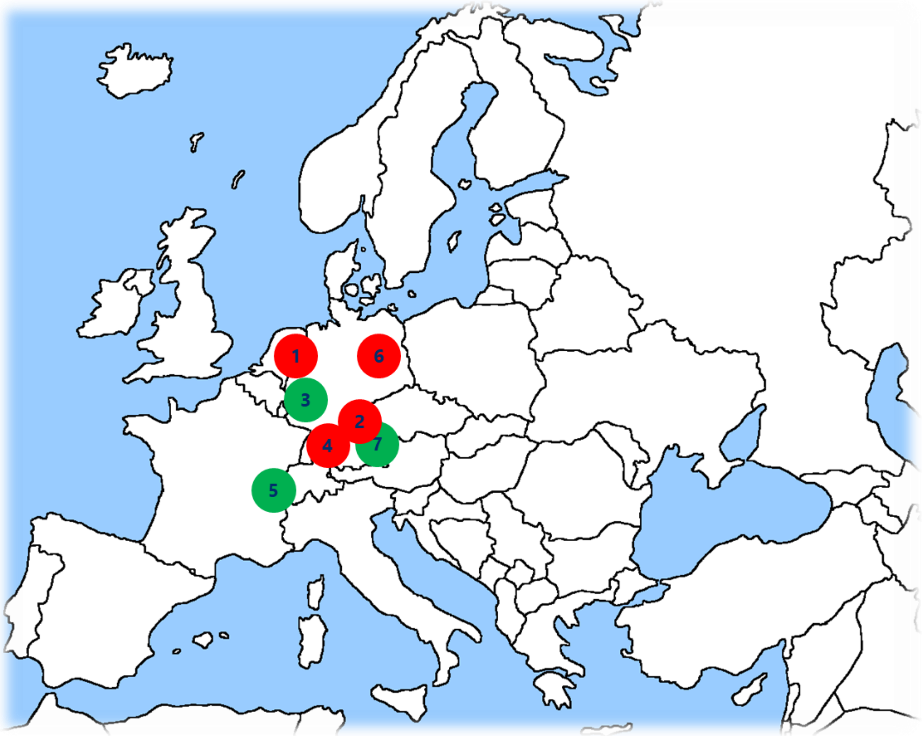
- host joint dialogues with organisations that have expert knowledge on materials testing at cryogenic conditions and handling of liquid hydrogen.
- determine the research capability, capacity, and programmes of work needed to develop competence in support of UK supply chains with emerging energy and environment technologies.
- identify potential opportunities for researcher exchange and bilateral training.

The outputs of this work are summarized in this open-access report, which is split into two main sections; the first describing the organisations visited in Europe and the second covering the organisations visited in the USA. In the Appendix, several collaborative research programmes, hydrogen related software tools and data, and additional information related to this work are detailed. It is anticipated that the information in this report will have a direct benefit for:

- Hydrogen projects within the UK tasked with determining the requirements for transitioning to cryogenic hydrogen, including HII SEED and the ATI's Hydrogen Capability Network (HCN).
- UK government by raising awareness as to the national underpinning infrastructure and research requirements needed to accelerate the transition to net zero and support emerging technologies and supply chains.
- UK industry sectors, including aerospace, which seek to develop cryogenic hydrogen technologies and require materials test and evaluation at realistic temperature and environmental conditions.

European landscape

Researching for capability and infrastructure for cryogenic and hydrogen testing in Europe, seven public and private organisations were identified as listed in Figure 1. Of those, three were prioritised to visit because of the maturity in materials level testing at cryogenic temperatures and in-situ cryogenic hydrogen conditions. The visits were conducted during the 11th-15th December 2023.



1. National Aerospace Laboratory (NLR) – Netherlands
2. German Aerospace Centre (DLR) – Germany
3. Karlsruhe Institute of Technology (KIT) – Germany
4. KRP Mechatec GmbH – Germany

1. European Organisation for Nuclear Research CERN – Switzerland
2. Federal Institute for Materials Research and Testing (BAM) – Germany
3. ET EnergieTechnologie GmbH – Germany

Figure 1: Identified and down selected European facilities with cryogenic and hydrogen test infrastructure.

Within the community of nuclear physics, the Materials, Metrology and Non-destructive Testing (EN–MME–MM) section at CERN (European Council for Nuclear Research) has developed a great deal of expertise and capability in the field of cryogenic testing. The main motivation behind this section's formation was that of supporting the constant need for understanding how materials operate over a range of temperatures, as is the case for a vast number of components involved in the operation of the accelerator experiments. They offer high-load mechanical testing capabilities at liquid Helium temperatures, as well as a full-suite of thermophysical characterisation, often utilising in-house one-of-a-kind equipment and instrumentation.

Notably, Karlsruhe Institute of Technology (KIT) have developed significant expertise in the field of cryogenics and hydrogen testing. The CryoMaK (Cryogenic Materials Karlsruhe) laboratory, within the Institute of Technical Physics, has amassed decades of technical expertise and unique capability for characterising materials at cryogenic temperatures, with the main motivation of supporting ITER [7] (international thermonuclear experimental reactor) and the nuclear physics scientific community. Currently, a wide range of mechanical and thermophysical testing is offered at different load levels and temperatures as low as 4 K, by utilising a centralised helium recovery and liquefaction system for the whole facility.

Finally, ET EnergieTechnologie are a privately owned company focusing on testing for hydrogen systems and the development of unique in-situ liquid hydrogen mechanical test capability.

CERN

Materials testing at CERN focuses on the demands of the high energy physics community who are developing experiments including particle accelerators and nuclear fusion reactors. CERN has world leading capability in cryogenic materials testing and cryogenic systems design and manufacture, and there has been significant investigation into the fracture of metallics.

The Mechanical and Materials Engineering group (EN/MME) has considerable mechanical and thermal testing capability. The 4 K mechanical testing capability consists of a wet LHe cryostat mounted on a mechanical test frame. There is also a second mechanical frame and cryostat awaiting commissioning which was designed and manufactured in-house.

The current system can test down to 4 K with a load capacity of 100 kN, with both quasi-static and fatigue (up to 50 Hz at RT) testing being possible.

For characterisation of thermal properties, the Mechanical Measurement laboratory of EN/MME's key areas of focus include dilatometry, the development of fibre optic sensors (to overcome issues around electromagnetic interference) and thermal diffusivity. An attoDRY2100 top-loading, closed-cycle cryostat (1.65 K to 300 K) is used with a range of interferometric probes for the measurement of thermal expansion (Figure 2). CERN have used both a commercial sample holder and developed their own custom sample holder to overcome some of the limitations of the commercial offering.

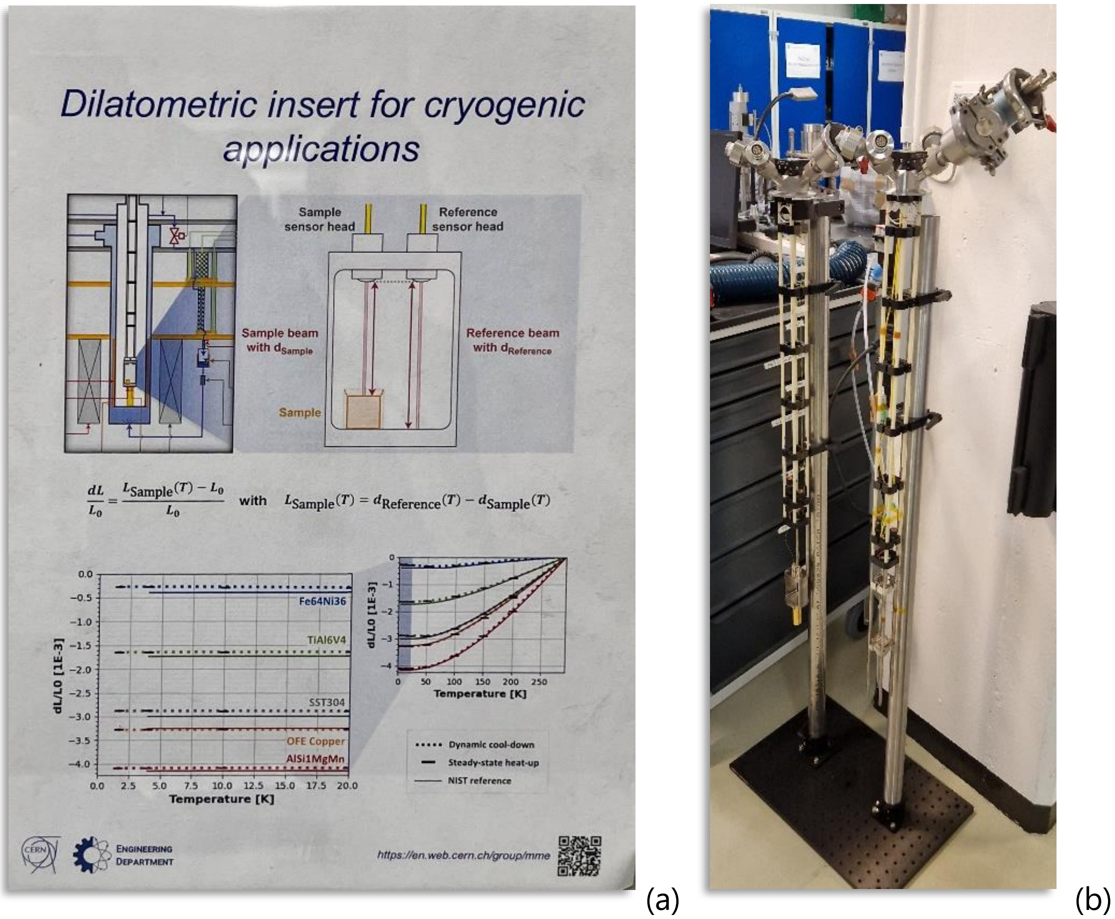


Figure 2 Dilatometric insert for cryogenic applications (a) principle of operation and measurements (b) set-up ready to be used in the lab.

Further, CERN has a suite of instruments for the measurement of thermophysical properties consisting of a Netzsch LFA 457 Microflash (-125 °C to 1100 °C) and a Netzsch LFA 427 Laserflash (ambient temperature to 2000 °C) for the measurement of thermal diffusivity, a Netzsch 404 DSC (ambient temperature to 1650 °C) for the measurement of specific heat and a mechanical dilatometer Netzsch DIL 402 E (ambient temperature to 1680 °C).

There is significant effort invested in the development of fibre optic strain measurement using fibre Bragg gratings to measure strain and characterise coefficients of thermal expansion. This is particularly relevant in the high energy physics community due to the high levels of electromechanical interference around superconducting magnets and close to circulating beams. For measuring strain during materials testing there is still a trend to use extensometers and traditional strain gauges, down to 4 K.

CERN have invested limited effort in internationally standardised methods due to bespoke and one-off nature of testing that is carried out in the high energy physics community. They have considerable interest in growing the cryogenic testing community to improve the range of capability and understanding of the fundamental science. Across CERN there is significant expertise and capability for designing and manufacturing cryogenic systems and so the design and fabrication of test equipment is often carried out in-house.

Karlsruhe Institute of Technology (KIT)

Work at KIT is focused on development of superconductor applications with a major topic towards Nuclear Fusion Magnet Technology but has now shifted to other sectors including hydrogen economy. There are several relevant research groups at KIT as listed below:

1) Cryogenic Material tests Karlsruhe (CryoMaK) – Part of the Institute for Technical Physics (ITEP) dedicated to materials testing and the focus of the visit.
2) Cryogenics Department - undertaking experiments for the Institute of Technical Physics (ITEP) and other institutes across KIT with cooling capacity and liquid helium. The department has a helium liquefaction, purification, and recovery system.
3) Institute for Thermal Energy Technology and Safety (ITES) – Modelling & Risk management of H_2 systems.

The EURATOM large coil task

Inside the northern campus of the Karlsruhe Institute of Technology (KIT) and next to the CryoMaK testing facilities stand two of the largest cryogenic “specimens” tested onsite. The EURATOM LCT (Large Coil Task) (pictured on the right) and ITER’s TFMC (Toroidal Field Model Coil) were delivered in Karlsruhe’s TOSKA facility ahead of their cold acceptance testing. The LCT coil was tested following the completion of the TOSKA facility back in 1984, while the TFMC coil was first tested alone in 2001 and then in 2002 within the background magnetic field of LCT.

KIT is a merger of the University of Karlsruhe which is funded by the federal state of Baden-Württemberg and the Helmholtz Large Scale Research Center receiving funding from both the German State Government as well as the European Union. KIT is a member of the Helmholtz Association, the largest research organisation in Germany, comprising 18 research centres totalling ~46,000 employees and an annual budget of 6-billion euros. In 2020, KIT’s budget was around 967.7 million euros. As of 2020, KIT employed nearly 10,000 people, more than half of which work in research, as some of the key collaborators included Helmholtz Association, ITER, CERN, DLR, EPFL, SIEMENS ABB, BASF, and AIRBUS, amongst others. ITEP and CryoMaK in particular, have long-standing ties with nuclear physics projects like ITER and therefore part of the funding for their relevant activities comes from such projects.

CryoMaK is a large cryogenic material testing laboratory with capability in both mechanical and thermal property measurements. For mechanical testing there is a large range of infrastructure including a range of wet bath and gaseous exchange cryostats. This equipment comes from a range of suppliers and some custom designs by their internal cryogenics department.

There is significant expertise in the group related to the development of both test fixtures and measurement systems and have designed, manufactured, and published details on a cryogenic extensometer including designs for measuring Poisson's ratio. The group has also implemented novel concepts for optimising the test procedure by means of faster cooldown and a multi-specimen loading system to enable several mechanical tests to be carried out in a single thermal cycle in a bath cryostat.

For LH_2 testing CryoMaK test both pre-charged specimens and test specimens in-situ with capillary tube centres, while setting up a LH_2 test capability in a CryoVac gaseous exchange cryostat. To enable this, the team are developing an on-site LH_2 liquefaction capability using LHe generated from their LHe liquefier.

Figure 3 In-situ LN2 test setup MTS100 at Karlsruhe Institute of Technology.

Expertise from the Institute for Thermal Energy Technology and Safety has also been integrated around H_2 safety and explosion modelling which they have used to enable the development of LH_2 testing onsite.

There is significant thermal & physical property testing capability using both wet and dry systems. Testing is carried out across a range of materials including metallics, ceramics, and fibre-reinforced plastic (FRP) composites. A detailed presentation can be found in [8] and [9].

Mechanical testing capability includes quasi-static, fatigue, fracture, and torsion. More specifically mechanical property measurement between 4.2 K – 300 K includes:

- ATLAS axial ±650 kN
- PHOENIX axial ±100 kN
- MTS 25/50/100 axial ±25 kN / 50 kN / 100 kN (Figure 3)
- MTS100 with LH_2 testing (to be commissioned)
- Torsion - combined axial (±100 kN) and torsion (±1,000 Nm) loading

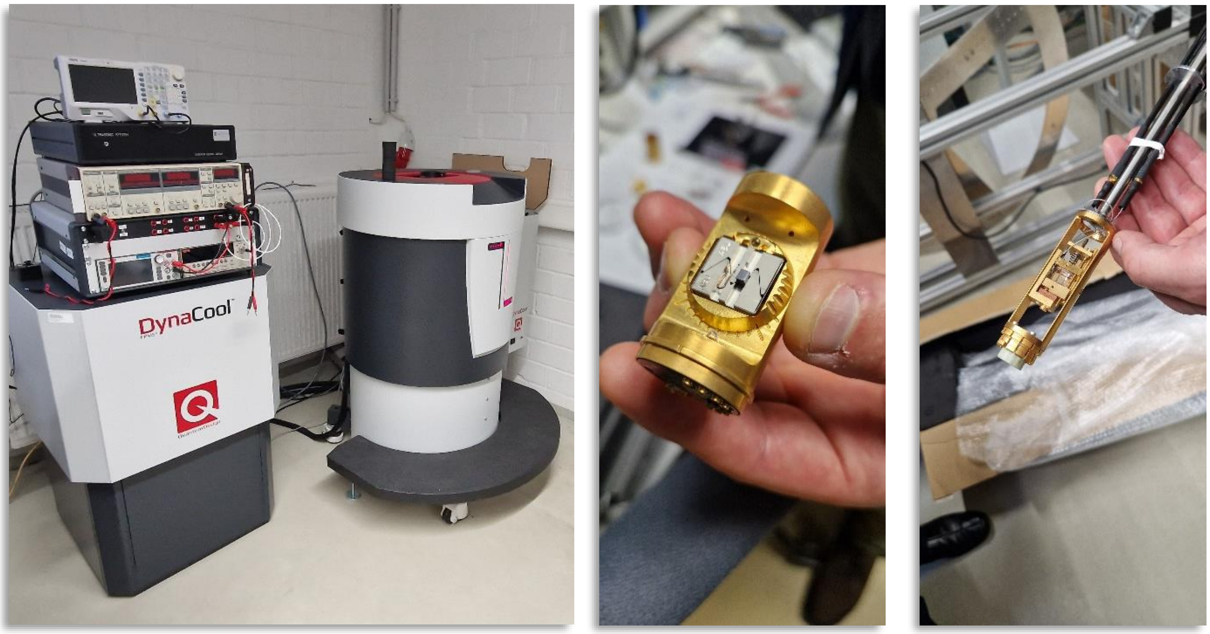


Figure 4 Quantum Design Physical Property Measurement System with thermal property probes.

In addition, the following measurements can be made in the same temperature range:

- Impact – two rigs are available including a Charpy (450 J) and drop weight tower, this testing is carried out by moving pre-cooled specimens into room temperature test environments.
- Hardness - Vickers test
- Electromechanical properties of superconductors (4.2 K-77 K, 100 kN, 12 T)

- The influence of mechanical loads in a magnetic field on the critical current of technical superconductors can be investigated with the FBI facility (F-force, B-magnetic field, I-current).

Two Quantum Design Physical Property Measurement Systems (PPMS) and a dry DynaCool System (2 K – 400 K, 9 T or 14 T) with a range of probes including:

- Thermal expansion,
- Heat capacity,
- Electrical and thermal conductivity,
- Magnetisation,
- Elastic Constants.

The cryogenic capability is coupled to extensive expertise in material analysis, failure analysis, spectroscopic analysis of the chemical composition of metallic materials (BRUKER Q4TASMAN), scanning electron microscopy (EDX/EBSD) and X-ray diffraction.

ET EnergieTechnologie

EnergieTechnologie are a small independent company with approximately 30 employees founded in 1997. The core focus is around testing for hydrogen systems and components. The main customers base has been driven by automotive, space and aerospace sectors with primary funding coming from commercial work.

EnergieTechnologie test across 3 core areas; hydrogen fuel cells, high pressure FRP gaseous storage tanks, and liquid hydrogen materials & products.

The focus of the visit was on the LH_2 testing facility where there are approximately two tons of LH_2 storage on site, used for both materials and product testing. Primarily the materials testing has focused on metallic materials while examples for product testing would be LH_2 storage tank systems, heat exchangers, etc. These tests may be done with liquid as well as supercritical hydrogen. Tank characterization usually involves filling and refilling processes, leakage tests, boil-off functionality, dormancy, and natural evaporation rates.

Materials testing has been available since 2016 and is carried out in a LH_2 bath cryostat mounted on a custom designed mechanical test frame with a maximum load capacity of 100 kN. This rig was designed in collaboration with the Universität der Bundeswehr München and ESA [10].

Figure 5 Liquid hydrogen testing machine at EnergieTechnologie.

Coupons as long as 200 mm can be accommodated, and the machine has a maximum travel of 50 mm. Both quasi-static and fatigue testing can be carried out with fatigue testing carried out at frequencies in the region of 10-30 Hz and loading above 1 kN and ideally in the region of 5-50 kN. Strain is measured using commercial cryogenic extensometers.

Tests can take from hours (tensile, fracture toughness) to days (fatigue), depending on the nature of the test. The test procedure involves mounting of the sample, closing, and conditioning of the container, cooling with liquid hydrogen, running the test (with refilling if necessary). After test end the system is warmed up and inerted.

EnergieTechnologie also offer immersion and temperature cycling of specimens in LH2.

ET is also able to perform tests with pressurized hollow samples (up to 100 MPa) and test in other media and at other temperatures (e.g., 77 K or ~200 K).

USA landscape

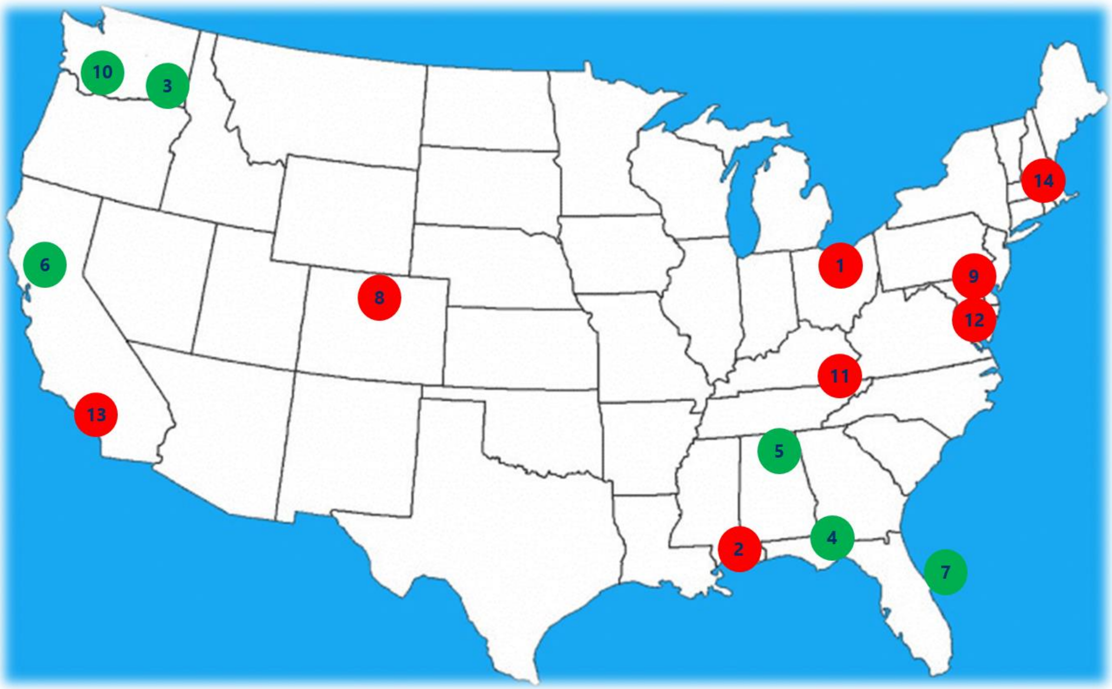
In early 2023, NPL explored what expertise related to thermal property measurement at cryogenic conditions, exists internationally and identified that the National Institute of Standards and Technology (NIST), the USA National Metrology Institute (NMI), had carried out a significant amount of work in this field. It was also understood that additional expertise and capability exists in materials characterisation in cryogenic and liquid hydrogen environments across the USA and at other national laboratories.

Following extensive literature and online research the organisations in Figure 6 were identified as being of interest for a visit. However, due to limitations on time, budget, and close alignment with the specific scope of this work, only visits, and bilateral meetings to the following six organisations were arranged:

1. Washington State University (WSU) in Pullman, Washington
2. Pacific Northwest National Laboratory (PNNL) in Richland, Washington
3. Sandia National Laboratories – Livermore, California
4. National High Magnetic Field Laboratory (NHMFL) and the Florida State University (FSU) in Tallahassee, Florida
5. NASA Marshall Space Flight Centre in Huntsville, Alabama
6. NASA Kennedy Space Centre in Merritt Island, Florida

The visits took place between the 11th - 24th February 2024 and detailed technical capabilities are presented in subsequent paragraphs of this report. In the interest of completeness, some key capabilities of the other centres in Figure 6 are summarised below.

The Creek Road Cryogenics Complex (CRCC) is a state-of-art facility at NASA's Glenn Research Center in Cleveland, Ohio. It comprises four separate test cells with unique capabilities. The Small Multi-Purpose Research Facility (SMiRF) evaluates the performance of thermal protection systems required to provide long-term, up to 10 years, storage of cryogenic propellants in space. The Cryogenics Components Lab 7 (CCL-7) is a smaller version of SMiRF with similar capabilities. The Cryomotor Test Bed is used to evaluate the performance of cryogenically cooled motors, and finally the 20 K to 90 K Calorimeter Test Bed is used to evaluate the performance of different insulation materials.



1. NASA Glenn Research Centre
2. NASA Stennis Space Centre
3. Washington State University
4. Florida State University
5. NASA Marshall Space Flight Centre
6. Sandia National Laboratories
7. NASA Kennedy Space Centre
8. NIST Boulder
9. Goddard Space Flight Centre
10. Pacific Northwest National Laboratory
11. Oak Ridge National Laboratory
12. Thomas Jefferson National Accelerator
13. NASA Jet Propulsion Laboratory
14. Commonwealth Fusion Systems

Figure 6: Identified and down selected United States based facilities with cryogenic and hydrogen test infrastructure.

NASA's Stennis Space Center in south Mississippi operates as the agency's primary, and America's largest, rocket propulsion test site [11]. Within Stennis Space Center the Cryogenic Storage and Transfer Facility maintains a fleet of cryogenic barges to move propellant to the A and B test stands.

The National Institute of Standards and Technology (NIST), in Boulder, Colorado, Cryogenic Technologies Group existed as a group from 1995 to 2009, running out of the Applied Chemicals and Materials Division. Some limited activities continue today divided into four areas: (1) education in the form of publications and short courses around cryogenics and its applications for use by the public and professionals, (2) cryocooler research, (3) cryogenic material properties database which constitutes a critical evaluation of existing experimental measurements on the properties of engineering materials at cryogenic temperatures, and (4) cryogenic flow calibration.

Goddard Space Flight Center is NASA's premiere space flight complex in Greenbelt, Maryland. The Materials Engineering Branch has extensive expertise in areas that include additive manufacturing, adhesive bonding, brazing & welding, ceramics & glasses, chemical analysis, composites, cryogenics, metallurgy, non-destructive evaluation, tribology, and others. The Cryogenics and Fluids Branch has developed expertise in thermophysical properties measurements (e.g., thermal conductivity and specific heat) as a function of temperature for materials spanning 4 K – 300 K utilising a cryostat-based system. The branch also has expertise in dewar design for both ground-based performance testing, as well as spaceflight systems.

Oak Ridge National Laboratory in Oak Ridge, Tennessee, is a Department of Energy (DOE) funded research centre and its scientific programs focus on materials, nuclear science, neutron science, energy, high-performance computing, environmental science, systems biology, and national security. The Mechanical Properties and Mechanics (MP&P) Group has a large collection of unique mechanical testing equipment with capabilities to conduct uni- and multi-axial tests from cryogenic to very high temperatures in air or controlled environments. The group specializes in the mechanical evaluation and characterization of structural and functional materials; in experimental mechanics and the development of test methods, design codes and life-prediction analyses; in establishing relations among processing, microstructure and mechanical properties of materials and structures and how these change as a function of time, stress, temperature, and environment. MP&M has expertise with a wide range of materials including metals, ceramics, polymers and glasses, metal, polymer and ceramic matrix composites, concrete, and functional materials (magnetic, electronic).

The cryogenics department in Jefferson Lab in Newport News, Virginia utilizes its specialized technical expertise to design, fabricate, and commission large cryogenic equipment at other facilities around the country including Oak Ridge National Laboratory, Michigan State University, Johnson Space Center, and Stanford University.

NASA Jet Propulsion Laboratory in Pasadena, California is a federally funded research and development centre administered and managed by the California Institute of Technology. The laboratory has expertise in thermal characterisation of advanced materials and most of the cryogenics' related activity is focused on chemistry at cryogenic temperatures and X-ray diffraction.

Washington State University (WSU)

Washington State University is a public land-grant research university in Pullman, Washington. Founded in 1890 it currently has approximately 25,000 students.

HYPER Lab was set up by Prof. Jacob Leachman in 2010 as a purely cryogenic hydrogen research centre. HYPER Lab is student led, has a community focused culture and averages between 20-40 people. At present the group comprises three academics Jacob Leachman (Head of Group), Konstantin Matveev (Cryogenic Fluids Behaviour) and Arezoo Zare (Materials and Mechanics) as well as nearly 20 researchers across undergraduate, graduate, and postdoctoral positions.

Most of the funding historically is via Government grants. However current funding is approximately 80% from private industry funding on a project-by-project basis. Some of the key collaborators include Airbus and Plug Power.

HYPER Lab performs liquid hydrogen research across multiple disciplines, focused on experimental testing with LH_2 and developing fluid dynamics and material models. More specifically the key research themes include:

- Materials (Test Methods, Multiphysics modelling)
- Safety (Handling protocols, Modelling, and experimental analysis of failure cases - Leakage, diffusion, Combustion)
- Thermofluids (Fluid properties, Multiphase flow, Spin Isomers)
- Test hardware development (Thermofluids testing hardware, Materials testing hardware)

The HYPER Lab team have a strong skills and safety culture with processes in place for peer-to-peer training and are currently setting up a “Cool Fuel School” for external training.

The team is currently performing tests by liquefying hydrogen gas in-situ. However, they are likely to have access to a 4.5 tonne tank which they plan to develop and enable the building of associated hardware through student projects. This tank is part of a LH_2 refuelling station proposed by Plug Power to enable decarbonisation of the heavy vehicle fleet at WSU as mandated by government legislation, a proposal that is currently awaiting a funding decision.

CRyogenic Accelerated Fatigue Testing (CRAFT) [12]

CRAFT allows for the tensile, compressive, and fatigue testing of polymer composites in a liquid nitrogen (77 K) or liquid hydrogen (20 K) bath (Figure 7). The facility consists of a fully electric load frame (MTS Acumen 12 – equivalent of Instron Electropuls E10,000 machine) capable of fatigue testing polymeric materials in a liquefied hydrogen environment (hydrogen gas liquefied in-situ). The static load capacity is 8.5 kN and the dynamic is 12 kN. The system was built and became operational in the Summer of 2020. To achieve vacuum, CRAFT uses a two-stage vacuum system. First, an Agilent Varian IDP-07 Dry Scroll Vacuum Pump is used to reduce the pressure to 2.5 Pa, and then an Agilent Turbo-V 81 M Turbomolecular Pump to bring the pressure down to ~10^{-5} Pa. For cooling, a Cryomech CP 1010 Helium Compressor combined with a PT415 Cryocooler are utilised.

Figure 7: The CRAFT facility for fatigue testing polymeric materials in a liquid nitrogen (77 K) or hydrogen (20 K) environment.

CRAFT has a steel body vacuum chamber to house the test environment; this is placed over the specimen and grip arrangement with the threaded lower pull rod screwed into the vacuum body base plate. Indium wire seals are fabricated in-house to ensure the vacuum body is effectively sealed at both ends. The test chamber can be filled with up to 0.57 litres of liquid. A custom bellows seal for translational movement is used to allow for the passage of the pull rod. CRAFT uses a thermal strap to move heat out of the system. Displacement is measured directly on specimen using a miniature Epsilon clip gauge extensometer. Temperature measurement is undertaken using silicon diode (Lakeshore) sensors. The facility has recently been used to characterise materials including polycarbonate that has been used in the creation of a flexible fuel bladder.

Cryo-catalysis Hydrogen Experimental Facility (CHEF) [13]

The use of liquid hydrogen for commercial applications can suffer from significant boil-off. The Cryo-catalysis Hydrogen Experimental Facility (CHEF) has been established to undertake research into minimising losses for terrestrial and space applications.

Orthohydrogen-parahydrogen conversion is the largest effective phase-change of any material at cryogenic temperatures from an energy or entropy standpoint. The Cryo-catalysis Hydrogen Experiment Facility (CHEF) was designed in 2011 to control the ortho-parahydrogen conversion of condensed hydrogen through careful material selection and catalyst implementation. The cryostat itself was retrofitted from WSU faculty members using it for plasma research. With a total liquid hydrogen capacity approaching 7 litres, CHEF has been the most heavily utilized cryogenic system in the first decade of the HYPER Lab.

The system is based on a cryocooler in the base of the unit, Cu coated tubes. The top can is lowered, sealed and then a vacuum is pulled. Uses a Raman cell to which optical fibres and spectrometer are attached to monitor wavelength of light and then measure peaks corresponding to ortho- and para- proportions of liquid hydrogen. Other properties are also sensitive to proportions of ortho- and parahydrogen including thermal conductivity, speed of sound, viscosity, melting points, boiling points, vapour pressures, and specific heat capacities. Density is unchanged.

The aim of the work is to enable standardisation of liquid hydrogen as a fuel by being able to measure proportions of ortho- and parahydrogen in a similar way to octane content in petrol.

Visualisation of vapour layer in liquid nitrogen using particle image velocimetry (PIV)

Studies being undertaken on vapour layer above liquid nitrogen in a polystyrene bath using laser light (5 mW) illumination and looking at differences between insulated and uninsulated states. The vapour layer is easily disturbed by hand. The lab is aiming to perform the same study for LH_2 to improve modelling of liquid-vapour state of LH_2 as currently errors in modelling are of the order of 50%. Plan to use a camera (at 2,200 fps) to record movement of vapour layer and then use particle image velocimetry (PIV) – analogous to DIC for fluids.

Thermal conductivity measurement of 3D printed MMCs

HYPER Lab have used ‘paddle’ shaped specimens to measure thermal conductivity of 3D printed Al composite (Al-ceramic-magnesium) heat exchangers. A cartridge heater is inserted into one end of the paddle sample whilst the flat end of the paddle is bolted to a cryocooler. Thermal conductivity is measured in the parallel sided section. Thermal conductivity can be tuned by changing the constituency of the material and minor changes in the processing route (print orientation). NIST used to have this capability but since older staff have retired some of the NIST experimental rigs have been passed to HYPER Lab.

Cubic Cryo Chamber (CCC)

CCC is a steel vacuum chamber designed to be easily configurable for cryogenic experiments.

Work done on permeation of hydrogen through polymer-based materials at low temperatures (100-150 K) has shown that permeation levels are equivalent to background hydrogen levels. At cryogenic temperatures everything slows molecularly so much so that permeation is essentially zero unless there is a pinhole leak. To date there has been no investigation done on damaged composites.

Re-design of pressure relief valves (PRVs) for H_2 applications

Pressure relief valves (PRVs) prescribed by API 527 (Seat Tightness of Pressure Relief Valves) are suitable for ‘conventional’ industries but not adequate for LH_2 applications. Foreign object debris (FOD), defined by Arizona dust standard (0.5 – 324 mm), can get caught between seal and seat and can cause leaks. HYPER Lab have performed work on re-designing of valves to have several seals fabricated from several layers of polymer membrane.

Reducing fill times for drone (GENII) liquid H_2 tanks

Key to use of hydrogen powered drones is rapid filling of liquid hydrogen fuel tanks. GENII drone was utilised for this project which has a maximum flight time of 2-3 hrs and older tank designs required about 1 hr to re-fill. With a use of multilayered wall structure of CFRP, nylon and glass beads, the tank fill time can be reduced to ~1 hr – the wall structure allows liquid hydrogen to ingress, boil off and reach a steady temperature state. Though, it is not known how much liquid is lost during this stage, however.

Hydrogen Thermo-acoustics work

HYPER has the CRATOS test stand which measures thermo-acoustic phenomena affecting liquid hydrogen. Temperature gradients between liquid hydrogen and ambient conditions lead to spontaneous pressure oscillations that pump heat from hot to cold, resulting in significant heat loads. CRATOS is characterizing these losses and inverting the effect for cooling.

Mobile Hydrogen Generation Unit (MHGU)

Initially a student project, a mobile hydrogen generation unit (MHGU) was developed consisting of an electrolyser, a cryocooler based refrigeration and an LH_2 dewar. The system can produce 60 litres of liquid hydrogen every 2 weeks (stored at 18 K). It runs on a 208 V supply or via diesel generator. Extensive research is currently underway related to H2 liquefaction.

In addition to the research activities, HYPER Lab plan to run a Cool Fuel School, a 2-3 weeks per year course that will qualify students to design hydrogen systems in a safe, systematic way.

Pacific Northwest National Laboratory (PNNL)

Pacific Northwest National Laboratory (PNNL) draws on its strengths in chemistry, Earth sciences, biology, and data science to advance scientific knowledge and address challenges in sustainable energy and national security. The scientific work focuses on advancing sustainable energy through decarbonization and energy storage and enhancing national security through nuclear materials and threat analyses. PNNL has twenty-two core capabilities recognised by DoE and organized into five areas:

1. Chemical and Material Sciences
2. Computational and Mathematical Sciences
3. Earth and Biological Sciences
4. Engineering
5. User Facilities and Advanced Instrumentation.

PNNL was founded in 1965 and is operated by Battelle for the Department of Energy's (DoE) Office of Science, which is the single largest supporter of basic research in the physical sciences in the United States.

PNNL's funding comes from DoE and industrial commercial contracts at approximately 50:50 ratio. The laboratory collaborates extensively with academia in fundamental research and with industry to transition technologies to market. In hydrogen, industrial partners come from land transport and battery energy storage industries.

PNNL employs 6,089 scientists, engineers, and professional staff (FY 2023) and its main facility is in Richland, WA with a second facility in Sequim, WA dedicated to Coastal Sciences. PNNL's research and development expenditure is approximately $1.5B (FY 2023).

Under the sustainable energy banner, PNNL is developing technologies for lighter weight and energy-efficient transportation, placing emphasis, amongst other things, on hydrogen and fuel cell research in the following key areas:

- Hydrogen Materials, where PNNL leads the development and dissemination of best practices for polymer materials hydrogen compatibility testing.
- Hydrogen Safety, with PNNL's hydrogen materials research feeding the Hydrogen Tools Portal, and providing key data about hydrogen properties, handling, and safety.
- Hydrogen Storage, where PNNL is involved in the Hydrogen Materials Advanced Research Consortium supporting the development of characterization tools and materials to advance hydrogen storage.
- Hydrogen Liquefaction, where PNNL is developing technology with the potential to double the efficiency of the hydrogen liquefaction process while lowering capital cost.
- Hydrogen Production, where PNNL is developing high-temperature electrolysis technology with significantly less electricity requirements compared to traditional low-temperature electrolysis. PNNL is developing processes for electrochemical and biological hydrogen production.
- Fuel Cells, where PNNL's work through The Institute for Integrated Catalysis focuses on the development of new electrocatalysts for low-temperature polymer exchange membrane (PEM) fuel cells and electrolysers.

The main motivation for the work that the materials team is involved in is physical hydrogen storage and in particular Type IV and Type V pressurised gaseous and cryogenic storage vessels. The team is involved in, amongst other projects, H2@Scale, the Hydrogen Materials Advanced Research Consortium (HyMARC) and the Hydrogen Materials Compatibility Consortium (H-Mat). Details of these programmes can be found in the Appendix.

Mechanical & Hydrogen Testing Laboratory

The mechanical testing laboratory is equipped with a continuous flow 10 kN rated liquid helium/nitrogen cryostat by Lake Shore Cryotronics on a 220 kN rated MTS servo-hydraulic test frame. The team is performing tensile tests on specimens based on ASTM E8 Standard Test Method for Tension Testing of Metallic Materials and short beam shear tests based on ASTM B2344 Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates. The cryostat uses a 100-litre liquid helium dewar managing up to 6 runs out of a single dewar by pre-cooling with liquid nitrogen to 77 K (~1.5 hrs) before pulling vacuum to get rid of the vapour and cooling to 20 K with liquid helium (~1 hr). Typically, the team tests one sample per day at 20 K or two samples per day at 77 K. PNNL do not have a helium recovery and re-liquefaction facility but their opinion is that it is well worthwhile if many tests are to be performed.

Tensile test fixtures are all made from Nitronic 60 stainless steel alloys as they retain ductility at low temperatures with higher strength compared to 300-series steels. A 310-alloy stainless steel short-beam shear (SBS) test rig produced by Wyoming Test Fixtures is used inside the cryostat with an additional alignment rig to maintain the relative positions of support and loading rollers, while self-weight of the pull rod and loading roller is used to maintain pre-load during cool-down. The set-up utilises an Epsilon extensometer with MIL-SPEC PT6 adaptor. Lake Shore Cryotronics provides thermocouples and calibration at cryogenic conditions. Temperature control is ±50 mK at the cryogen vaporizer. The vertical temperature gradient across the sample chamber is less than 0.5 K.

Further to mechanical testing at cryogenic temperatures, the laboratory has extensive test capabilities for in-situ hydrogen testing. These include:

- In-situ H_2 creep rig used to dead load specimens in a 3-point bending configuration and utilises digital image correlation (DIC) to measure the deflection.

The system is fitted with a window to enable observation of crack growth. (Figure 8)

- Thermal desorption analyser capable of quantifying the amount of hydrogen as it desorbs from soaked material.
- Permeation rig used to evaluate the permeation and diffusion coefficient of thin films and utilises a porous ceramic sinter to support the samples. The system is connected to an INFICON Micro GC Fusion Gas Analyser.
- In-situ DMA under pressure/gaseous environment, which has been used successfully with helium gas.
- High pressure 14,000 psi (~ 1000 bar) hydrogen enabled swelling of polymer gasket/seal discs with viewing window to perform 2D digital image correlation measurements to image local strains.
- Four extra small volume (5 ml) 15,000 psi (~ 1000 bar), four small volume (0.5 litres) 13,000 psi (~900 bar), two medium volume (3.7 litres) 4000 psi, and one large volume (8 litres) 4,000 psi (~275 bar) hydrogen rated pressure vessels for static exposure of materials.
- In-situ reciprocating tribometer for wear tests in H_2, He and Ar gasses (velocity of the wear head is up to 50 mm/s, maximum normal load 39 N, maximum temperature 50 °C and 4000 psi pressure of gas).

The laboratory is primarily equipped with hydrogen rated systems from HiP High Pressure Equipment (e.g., valves, fittings, tubing) and Haskel (e.g., pumps).

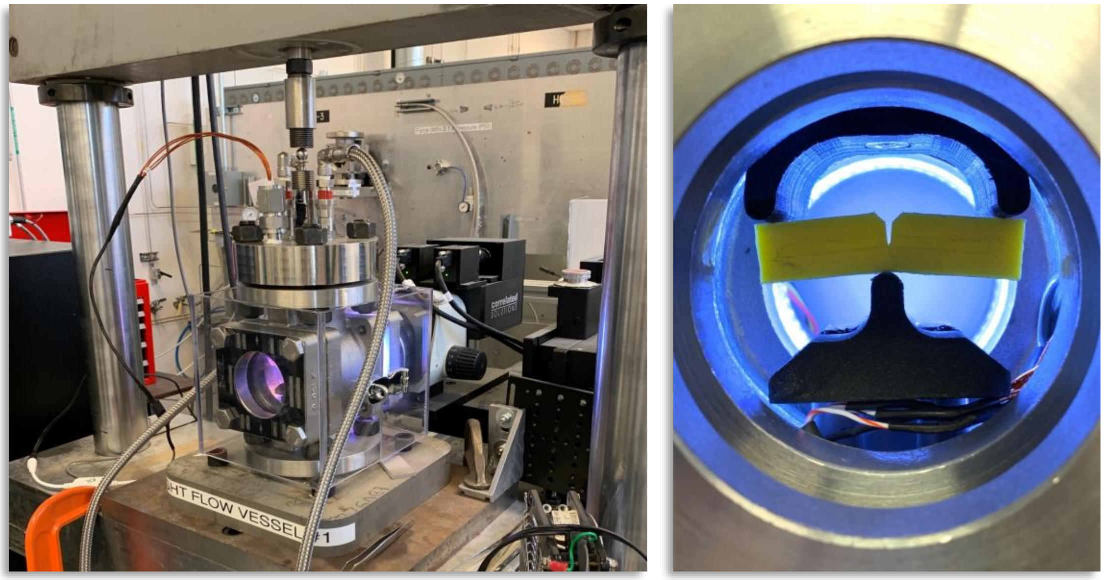


Figure 8 In-situ fracture test system at PNNL.

The hosting team discussed the potential of close collaboration with NPL, and other UK based organizations through Visiting Researcher positions and opportunities. For this to materialize an agreed and signed Memorandum of Understanding (MoU) will enable open discussions and information exchange.

Another mechanism for engagement that was discussed was that of the Technical Groups within VAMAS where organisations can openly share technical progress and conduct pre-normative research for the advancement of materials metrology.

PNNL's key partners include Sandia National Laboratories, National Renewable Energy Laboratory, Lawrence Livermore National Laboratory, Lawrence Berkeley National Laboratory, SLAC National Accelerator Laboratory, and National Institute of Standards and Technology, Argonne National Laboratory, Savannah River National Laboratory and Oak Ridge National Laboratory.

Sandia National Laboratories

Sandia National Laboratories (SNL) is operated and managed by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc. National Technology and Engineering Solutions of Sandia operates Sandia National Laboratories as a contractor for the U.S. Department of Energy's National Nuclear Security Administration (NNSA) and supports numerous federal, state, and local government agencies, companies, and organizations.

From the 1960's, SNL has had a core mission to support the nuclear deterrent program in the USA and development of enabling science for energy programs. At SNL, decades of hydrogen research and development has been undertaken since then with a focus on materials and safety. SNL provides deep, quantitative understanding and a scientific basis for materials for hydrogen production, storage, delivery, and utilisation, as well as risk analysis and the creation of risk-informed standards.

As a Federally Funded Research and Development Centre (FFRDC), Sandia can undertake work for industry if it responds to certain types of federal government solicitations. The solicitation must allow FFRDC participation and meet the requirements of Sandia's management and operating contract with DOE/NNSA. Sandia is also able to collaborate with industry via a mechanism called CRADA (corporate research and development agreement) which requires a 20% cost share from industry.

Sandia employs approximately 12,200 people, mostly at its headquarters in Albuquerque (New Mexico) or its second principal laboratory in Livermore, California. Other employees work at various sites in the U.S. and abroad.

A key focus for Sandia is looking at materials compatibility, and particularly the understanding of hydrogen embrittlement phenomena at atomistic scales. Hydrogen embrittlement occurs in materials under the influence of stress in hydrogen environments and as such the research activities of the Hydrogen Effects on Materials Laboratory (HEML) are focused on the combination of materials (strength, microstructure, chemical structure), environment (partial pressure, impurities, thermodynamics) and stress state (damage evolution, kinetics). Research themes cover:

- Surface interactions between hydrogen and materials,
- Transport and trapping of hydrogen,
- Rapid gas decompression,
- Hydrogen-assisted fatigue and fracture,
- Hydrogen effects on deformation,
- Mechanisms of fatigue and fracture,
- Evolution of damage.

The HEML also has a safety codes and standards programme which is an enduring programme and is annually reviewed and funded.

Sandia is currently or has been involved in several hydrogen research projects i.e., HydroGEN, HyMARC, H2FIRST, HyBlend, H-MAT, SHASTA and eXtremeMAT, details of which can be found in the Appendix.

SNL have a core capability for performing tests on materials in a hydrogen environment in the Hydrogen Effects on Materials Laboratory (HEML).

High-pressure H_2 testing

HEML have two test cells each capable of test pressures in gaseous hydrogen up to nominally 1,060 bar. One cell is used for room temperature measurements in gaseous hydrogen and the other cell is used for tests with temperature control (nominally between -40 °C and +100 °C) in gaseous hydrogen. An accumulated pressure of hydrogen is maintained for each cell above the test pressure to overcome any leaks through the seals thereby maintaining the supply of hydrogen at an ideal pressure. Testing in a gaseous hydrogen environment is done inside an autoclave on MTS mechanical test machines.

Fatigue crack growth (FCG) measurements using compact tension specimen geometries are performed in the high-pressure cells. Sandia staff perform FCG measurements by controlling the stress intensity factor, K, and therefore each test can sweep through specified R ratios using a single coupon [14]. On conclusion of each fatigue test, a fracture test is often performed on the coupon. Displacement and load are monitored using a crack opening displacement (COD) extensometer and an internal load cell, respectively. Rather than using a strain gauge-based load cell which would be affected by the hydrogen environment, the Sandia team use an LVDT-based proof ring to measure load on the specimen, internally. The benefit of using an internal load cell is that load measurements are not affected by the issue of friction between the load-chain seals and the pull-rod. Effects on fatigue and fracture can be seen at pressures as low as 1 bar. Testing can be undertaken using 3% hydrogen in nitrogen gas, which is considered a non-flammable gas at this concentration; however, most testing is performed using 99.9999% hydrogen source gas.

The Sandia team also test tubular specimens, fabricated from stainless steel, in which holes are drilled to act as an initiation site for crack growth. Direct current potential difference (DCPD) is used to monitor the crack initiation and growth. This type of specimen can only be used in tension-tension fatigue as the length of the pull rods mean that alignment can be an issue which can result in buckling of the specimen. Sandia do not have any concerns over using electrical devices in a pure hydrogen environment as the lack of an oxidizer in a well-controlled hydrogen environment prevents combustion. This contrasts with the use of electrical devices in hydrogen environments in countries such as France or Japan where this practice is banned.

The purity of hydrogen gas used is very critical as the presence of small amounts of oxygen can influence measurements; the current standard impurity limit is 2 parts per million of oxygen (CSA CHMC1), but this is likely to be revised to one part per million as even at this level measurements can be affected. Impurity measurements are made on samples of gas captured from the test vessel to ensure low impurity contents of moisture and oxygen.

A special manifold system has been designed and implemented for supply of gases to the high-pressure test cells. The system has 3 purposes: (1) to purge the test cell, (2) to maintain the desired gas pressure, and (3) to enable verification of the gas concentration. The system is automated and controlled from outside the test rooms. A robust procedure for purging the test autoclave to remove impurities prior to testing has been implemented, consisting of a minimum of three purges with N_2 at 140 bar and three purges with H_2.

Sandia conducts high-pressure gaseous hydrogen tests on subscale pipe coupons (surrogate coupons to replace full-scale tests) which are approximately 300 mm in length and 60 mm in diameter (see ASTM A53[15]). Internal defects are created in the pipe walls using a plunge EDM machine; external defects are created by the same methods with the aim of testing an undisturbed internal mill scale. Strain on the outside of the pipe is measured using a biaxial strain gauge located near the internal defect but on the outside of the pipe. The pipe samples are unconstrained axially, but the internal pressure is maintained due to the seal between the endcaps and the pipe. Hydrogen gas is fed in via the bottom end fitting. Typically, each pipe sample is pressure cycled to failure at a rate of one cycle per minute.

For polymers, various specimen geometries are used, and the materials are examined following exposure to repeated pressure cycles. The pressure range, temperature and quantity of pressure cycles is varied to examine the influence on damage, crystallinity, void formation, and swelling.

Hydrogen pre-charging can be performed within primary and secondary pressure containment vessels up to 1,400 bar and 300 °C. Specimens are held for a set time to achieve saturation; this is typically 60 days if conditioning a 12 mm thick compact tension specimen (or more typically 10-14 days for a 4mm diameter tensile specimen). When using autoclaves for pre-charging the internal volume of the vessel can be minimised with metal specimen holders, slugs, or steel ball bearings.

Low-pressure H_2 testing

Low pressure H_2 work is also conducted focused on measuring gas permeation through materials 25 mm in diameter and 0.25 to 1 mm thick. Gas-phase permeation is conducted under low-pressure (1 atm - 10^{-8} torr vacuum on downstream pressure) using deuterium to improve resolution of measurements (H_2 permeation) as there always tends to be a high background level of hydrogen. Permeability, diffusion, and solubility can be extracted from these permeation tests. The seals for the permeation system at SNL are copper gaskets for high temperatures, whereas Savannah River National Laboratory uses knife edge seals. For stainless steels, the permeation rates tend to be very slow so the way to increase the permeation rate is to raise the temperature and use very thin samples. Permeation experiments can be conducted at pressures just above 1 atm as pressure does not influence permeation rate or diffusion coefficients.

Sandia also have a low-energy ion scattering spectroscopy (LEIS) system which is a unique capability for observing how H_2 attaches to the surface of materials. X-ray photoelectron spectroscopy XPS is also used to analyse the surface kinetics of H_2 uptake and surface reactions.

The outputs of Sandia's research are made freely available as journal and conference papers as well as Sandia reports. Most content is available through the Office of Scientific and Technical Information (OSTI). Details on some of these tools and Resources can be found in the Appendix.

The work that Sandia undertakes on fatigue and fracture at coupon level in gaseous hydrogen environments is being conducted to develop and validate the use of Codes such as ASME Code B31.12. The work is focused on understanding of fatigue crack growth rates for various American Petroleum Institute (API) pipeline grades used for hydrogen, which, when combined with testing at NIST-Boulder and the open literature, provide the basis for design curves accepted into ASME B31.12 Hydrogen Piping and Pipelines code for fracture mechanics-based design. To date, Sandia has undertaken tests on dozens of vintages (i.e., materials that have been in service for several years) and modern materials.

The work at Sandia has shown that when ferritic samples were removed from a hydrogen environment the fatigue crack growth rates revert to the rates observed under a standard laboratory environment. For certain metals pre-charging in hydrogen can maintain the effect of hydrogen on the material even in an oxygen rich environment. For example, for austenitic metals the rate at which the hydrogen comes out of pre-charged material is much lower so there might be less of a need to do the test in a hydrogen environment, depending on the questions testing tries to address. Pre-charging is effective for comparative tests, but testing in gas for austenitic stainless steels and nickel-based alloys may still be needed for generating design data. However, one must also consider the kinetics of hydrogen uptake. For example, SS A286 often shows no effect during tensile testing in H_2 gas but shows substantial effects in long-term gas tests and when H_2 pre-charged.

Other related discussion points during the visit included:

- Some work done at SNL on hydrogen release utilising imaging with laser light, for composite vessels at low temperature.
- SNL have done work on H_2 release (not materials focussed) for building codes Early work was performed with high pressure H_2 release using liquid helium to generate small quantities of liquid H_2. Work undertaken has focused on safety codes and standards and understanding flammable releases e.g., what are safe standoff distances for other buildings from hydrogen storage facilities.
- In late January 2024, SNL performed a study looking at dumping of liquid hydrogen on different surfaces. This work will be soon available in the public domain.
- In Livermore there is a commercial transfer facility for liquid hydrogen; H_2 fuel cell vehicles commercially sold and there are filling stations in the bay area.
- Hydrogen fuel cells are now being used to replace diesel generators for producing power for lighting for road works. These mobile lighting units have been used also for the Oscars ceremony.
- The current push for hydrogen fuel applications is for heavy duty trucking e.g., Plug Power. Expectation is for liquid hydrogen and high-pressure gas fuel sources to become prevalent.
- Fuel cells are being developed to power barges to transport foodstuffs around Hawaiian Islands using liquid hydrogen power.
- ASME Pressure Vessels and Piping Conference has seen a much larger attendance and number of submitted papers focused on materials testing for hydrogen in recent years. In parallel, a study group meeting organized by SNL (for almost 10 years) has been associated with the ASME conference venue in recent years but remains independent of the conference.

Key collaborators include, but not limited to: Idaho National Laboratory, Lawrence Livermore National Laboratory, Berkeley Laboratory, National Renewable Energy Laboratory, Pacific Northwest National Laboratory, National Institute of Standards and Technology, SLAC National Acceleratory Laboratory, National Energy Technology Laboratory, Oak Ridge National Laboratory, Savannah River National Laboratory, Los Alamos National Laboratory, The Ames Laboratory.

National High Magnetic Field Laboratory (NHMFL) and the Florida State University (FSU)

The National High Magnetic Field Laboratory (NHMFL or MagLab) is funded by the National Science Foundation (NSF) and the State of Florida. It hosts the world's most powerful magnets to serve users worldwide. The MagLab has seven user facilities located across three campuses in Tallahassee (steady state magnets), Gainesville (hi B/T magnets) and Los Alamos (pulsed magnets). Every year more than 1,800 researchers use the MagLab's facilities and publish more than 400 peer-reviewed publications.

The NHMFL employs approximately 700 people, with 40% being students or post-doctorate researchers.

The work undertaken at NHMFL is interdisciplinary research combining chemistry, physics, and material science. Specific research themes include:

Condensed matter science: Complex behaviour near metal-insulator transitions, design and synthesis of novel materials, exotic superconductivity and other emergent ground states, low-dimensional electronic structures, quantum information technology, and quantum magnetism.

Cryogenics: Focus lies in the realm of cryogenic helium, particularly superfluid helium. The Cryogenics Group is at the forefront of developing advanced flow visualization techniques to explore the intricacies of turbulence and heat transfer in liquid helium. In addition, research covers helium-based dark matter detection, accelerator cryogenics, and quantum-fluid-based qubit platforms. A recently launched collaborative project, funded by NASA, has been established to conduct R&D work pertaining to liquid hydrogen-based aviation systems. The basic concept revolves around harnessing LH_2 to cool superconducting power system components, subsequently routing the warmed GH_2 to fuel cells. Within this initiative, the cryogenics lab is responsible for the design of a liquid hydrogen storage and transfer system together with heat exchangers to ensure the requisite LH_2 fuel flow rate as well as the delivery of the required cooling power to each power system component. In the past, the laboratory has worked on thermal conductivity measurements of MLI blankets and other insulation materials down to 20 K for NASA and industrial partners.

Geochemistry Group: The geochemistry group's research is centred around the use of trace elements and isotopes to understand Earth processes and the environment in the broadest sense.

Electro-mechanical Testing Group

The core capability of interest and relevance to materials characterisation at cryogenic temperatures is that contained within the Electro-mechanical Testing Group which serves the needs of magnet R&D projects. The Group carry out independent research and collaborate with national laboratories and industry.

The Electro-mechanical Testing laboratory can perform tensile tests at room temperature, 77 K, 4.2 K in a load range up to 500 kN (Figure 9(a)). A large amount of testing is undertaken on superconducting wires and mechanical properties of steels at cryogenic temperatures. The facility is fitted with a helium recovery system that has an efficiency of >95% thereby drastically reducing the net cost per litre of liquid helium.

Work in the Group is also focussed on undertaking critical current test measurements on superconducting materials (N.B. the critical current in a superconductive material is that above which the material is normal and below which the material is superconducting, at a specified temperature and in external magnetic fields). Within a cryostat, the test specimen is gripped, strained and current is flowed through the sample with the test operator looking for when the resistance becomes non-zero to define the critical current.

Mechanical measurements down to 77 K are undertaken on a mechanical test frame fitted with a cryostat which is filled with liquid nitrogen. Metallic samples, which are typically waisted to promote failure within the gauge section and reduce the magnitude of failure loads, are gripped using a set of lateral grips with serrated faces and a series of bolts are torqued to apply adequate gripping pressure. The problem with this type of grip is that if the loads required to fail a sample are too high then the thread on the bolts used to hold the grip faces together can be stripped off through the application of excessive torque. Usually tests at room temperature are successful, but for low temperature tests problems have been encountered with the grips not functioning correctly through loss of clamping pressure. Cryogenic rated grips from Wyoming Test Fixtures had been sourced by the laboratory (fabricated from 300 series stainless steel with tungsten carbide wedges).

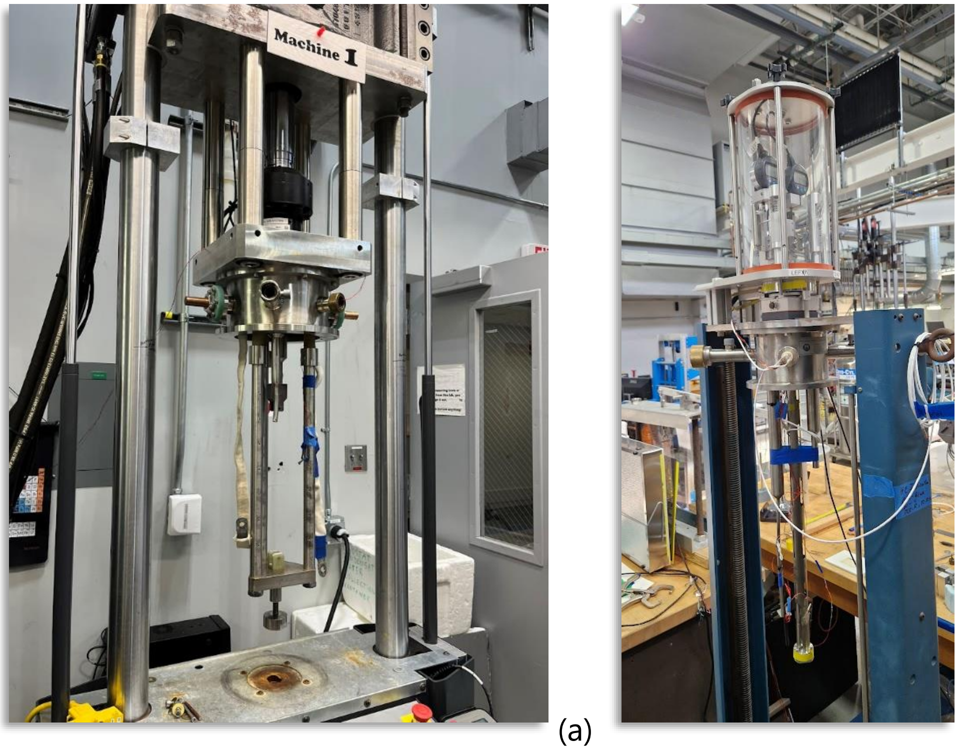

(a) (b)

Figure 9 (a) Liquid N_2/He cooled cryostat on a mechanical test frame and (b) Cryogenic dilatometer probe capable of measuring thermal expansion to 4K.

The laboratory is equipped with a Quantum Design Physical Property Measurement System (PPMS) which can be used to perform thermophysical tests from 4.2 K to room temperature (magnetic field 0 to 9 T). The system can be used to measure specific heat capacity (Cp) as a function of temperature (gold used as a reference material) and using the Thermal Transport Option (TTO) a sample's thermal conductivity, Seebeck coefficient, and electrical resistivity can simultaneously be measured as a function of temperature although this is not very accurate above 200 K.

The team also utilises a low temperature dilatometer suitable for coefficient of thermal expansion (CTE) measurements down to 4.2 K (Figure 9(b)). This equipment was built in-house and was adapted from a high temperature dilatometer system. The manufacturer of the high temperature dilatometer system was Anter Laboratories which are no longer in business. For cryogenic use, the quartz guide tube was exchanged for a titanium version that can accommodate two specimens: one a reference copper specimen and the other being the test specimen. The measurement part of the dilatometer, consisting of a pair of digital micrometers, is thermally isolated from the cold part using a water collar. This keeps the measurement head near to room temperature which maintains measurement stability (particularly as the laboratory temperature can fluctuate). The bottom of the micrometre gauge plungers is curved in shape and match up with dished indentations on the top of the reference and measurement samples. A level detector (sourced from a company called American Magnetics) is used to maintain an initial level of liquid helium sufficient to cover the test and reference samples. Three silicon diode temperature sensors are placed in the locality of the test specimen, and these agree well during the test. The system can be set running and left overnight; the helium gradually evaporates therefore the CTE measurement can be made over the range of 4.2 K to room temperature.

The low temperature dilatometer has been used to measure the CTE of coatings; the coating was applied to a series of steel blocks the CTE of which was then measured. By subtracting the known CTE of the steel the CTE of the coating material can then be found.

NASA Marshall Space Flight Center (MSFC)

NASA's Marshall Space Flight Center (MSFC) is located on the US Army's Redstone Arsenal in Huntsville, Alabama. The focus of MSFC is on ensuring the success of space missions with work focused on materials and engineering for space station modules and propulsion systems (failure analysis and material diagnostics), as well as understanding the effects of space flight environmental regimes on materials.

NASA MSFC has an annual budget of approximately $5 billion which is approved by Congress. Part of MSFC's portfolio involves working with commercial organisations, e.g., performing acceptance testing for space exploration companies. A percentage of this work is undertaken by MSFC's hydrogen materials test laboratory is commercial.

NASA MSFC has approximately 7,000 employees, of which 50% are employed by NASA and 50% are contractors. Approximately 400 staff work in the materials laboratory.

While NASA Langley and Glenn's work is more research based, MSFC is focused on application. Current work is aimed at supporting propulsion systems for future manned Lunar and Mars missions. Work on hydrogen is targeted at how to contain and sustain volumes of LH_2 prior to and during launches.

Materials and Processes Laboratory

The Materials Mechanical Test Facility (MMTF) has the capability to run tests at room temperature, at temperatures equivalent to the boiling point of liquid nitrogen (77 K) or temperatures that can be achieved via liquid nitrogen evaporative cooling. Additional LN_2

test work is undertaken at the Hydrogen Test Facility (HTF) using gaseous or liquid hydrogen.

The MMTF has a mix of approximately 25 electro-mechanical and servo-hydraulic test frames which are utilised for rudimentary test campaigns. A significant amount of work is being conducted on characterising the performance of thermal insulation foams e.g., compression performance at -179 °C (-290 °F) using evaporation of liquid nitrogen within a conventional test chamber. In addition, foams were being tested in compact tension (stress intensity factor), using digital image correlation (DIC Aramis - Carl Zeiss GOM metrology) to track the crack position. MSFC staff member has developed a method for applying a suitable speckle pattern to the foam blocks for this test.

The laboratory has the capability to undertake fatigue crack growth (FCG) measurements in liquid nitrogen on clevis attachment components. These tests were variable amplitude fatigue based on the spectral load profile endured during launch/flight; the tests typically last for~16 hours. Specimens are immersed in liquid nitrogen using a single walled 'bucket' cryostat sprayed with a foam that is used for insulation on rockets. The liquid nitrogen requires topping off every 1 hour or so.

Tests can also be undertaken in a LN_2/LHe cryostat which consists of an outer jacket that can be filled with LN_2 and an inner chamber that can be filled with LHe. This design allows for cryogenic testing to be done at LHe temperatures while significantly reducing the evaporation of LHe.

Hydrogen Test Facility (HTF)

High pressure gaseous and liquid hydrogen testing is undertaken in the Hydrogen Test Facility (HTF), which is a purpose-built facility situated in an isolated part of the MSFC site. The building has dedicated fire protection and hydrogen detection systems that cost ~$1 million to install. Over 90% of the work undertaken by the HTF is on metals and 80% of the work is for commercial contracts.

The HTF is equipped with 3 high pressure gaseous hydrogen test stations (9,600 psi / 662 bar), 7 uniaxial liquid hydrogen cells ranging in load capacity from 5,000 lbs (1,125 N) to 100,000 lbs (444 kN), and a single multiaxial test rig that has been used for permeation testing. All the test stations are situated outside of the control building. The facility undertakes a significant amount of fatigue crack growth (FCG) testing in either gaseous or liquid hydrogen environments. For FCG tests conducted in low growth (low da/dN) threshold regimes, load trains are sealed via the use of specialist bellows and load cells are kept outside of the hydrogen environment at approximately room temperature. The HTF is continuously having to replace equipment because of the effects of hydrogen on materials.

For studies on hydrogen embrittlement, the laboratory can pre-charge in H_2 and then store specimens in LN_2 before being tested. Sometimes samples are pre-charged in H_2 and then tested in a hydrogen environment. The maximum temperature that materials can be tested at is (2,000 °F, ~1,100 °C).

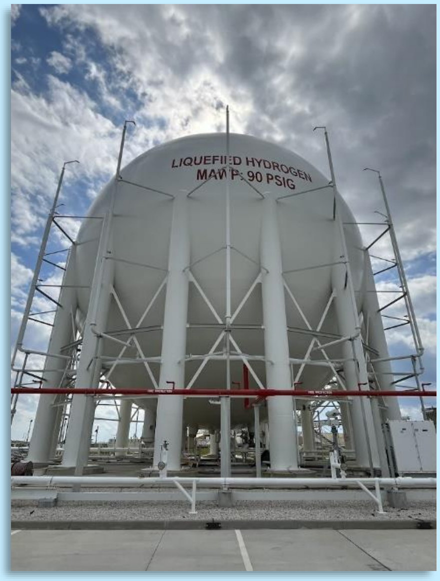
The HTF has a liquid hydrogen tank of 2,650-gallon (~10,000 litres) capacity which is filled ~4 times per day. The facility uses on average ~10,000 gallons (~37,800 litres) of liquid hydrogen every day. Using liquid hydrogen, a single tensile test can take typically 5 hours. On completion of the test, liquid hydrogen is left to boil off before nitrogen is flowed through the cryostat as a purge gas during warm up. Purging used to be performed with helium, but this is now unaffordable.

NASA staff commented that to build a facility such as MSFC's HTF would cost ~$20 million on infrastructure alone. However, the hard part is to find staff with the sufficient expertise and knowledge to run the facility. The requirement to have a minimum of two staff members (at least one of which must be certified) always on site (24/5) can also be a challenge. There is significant demand for the work undertaken by the HTF, and NASA say that they could easily double the number of test frames that they currently have.

NASA Kennedy Space Centre (KSC)

The National Aeronautics and Space Administration (NASA), an independent agency of the U.S. Federal Government, was established in 1958 and began operations to support civilian research related to space flight and aeronautics. The Kennedy Space Flight Centre was established in early 1960s as NASA's Launch Operations Centre by acquiring land on Merritt Island in central Florida and developing plans for Launch Complex 39 (LC-39) facilities which include the Launch Control Center, Pads A & B as well as the Vehicle Assembly Building (VAB). The Kennedy Space Centre consists of about 700 facilities and buildings.

New liquid hydrogen sphere at NASA KSC



The world's largest LH_2 storage tanks were constructed in the mid-1960s at NASA Kennedy Space Center by Chicago Bridge & Iron. These were vacuum perlite insulated tanks 3,200 m^3 in capacity and are still in use today. In 2019, Chicago Bridge & Iron began construction of an additional 4,700 m^3 LH_2 storage tank at LC-39B to support future missions, as NASA's new Space Launch System (SLS) heavy lift rocket for the Artemis programme holds 2,000 m^3 of LH_2 in its flight tank. For this tank, new energy-efficient technologies were implemented. An evacuated glass bubbles insulation system (passive control) has been realized as this technology has shown to reduce LH_2 boil-off by 46% when compared to perlite. In addition, an internal tank Integrated Refrigeration and Storage (IRAS) heat exchanger was employed to enable future full control/zero boiloff and/or densification of LH_2.

As a federal agency, NASA receives its funding from the annual federal budget. The Kennedy Space Centre has supported NASA Programmes such as Apollo, Skylab, Space Shuttle, Constellation, and currently, Artemis. Today, the Kennedy Space Centre, in conjunction with the adjacent Cape Canaveral Space Force Station, is a multi-user spaceport, facilitating the world's largest concentration of space launch operators.

Over the years, NASA and the Kennedy Space Centre have driven the development of large-scale liquid hydrogen (LH_2) storage solutions to serve the launch of various space vehicles. The Cryogenics Test Laboratory (CTL) supports Kennedy Space Center processing and launch activities and focuses on research and development in thermal insulation systems for both terrestrial and space applications, cryogenic component testing, next-generation cryogenic propellant storage and densification and low-temperature applications.

Since the completion of Launch Complex 39 in the 1960s, cryogenic technology has been progressing to accommodate the spaceport hydrogen operations. These need to be optimized for large quantities of LH_2, very unsteady demand and strict delivery requirements. Due to the high cost of LH_2, various projects have been funded to increase the efficiency of LH_2 storage and transfer. Between 2002 to 2006, NASA and the Department of Energy funded a lab-scale proof of concept for Integrated Refrigeration and Storage (IRAS), followed by IRAS heat exchanger characterisation tests in 2008/09. More recently, the Ground Operations Demonstration Unit for Liquid Hydrogen (GODU-LH_2) project (2012 – 2016) was funded aiming to demonstrate, on a medium scale (125 m^3), advanced operational concepts using IRAS that would reduce liquid hydrogen losses and increase ground control of cryogenic propellants in support of launch vehicles. Additionally, from 2004-2007 the CTL matured glass bubbles insulation for LH_2 tanks through the New Materials & Technologies for Cost-Efficient Cryogenic Storage & Transfer (CESAT) project; the results of which were key in convincing NASA to implement the technology in the new 4,700 m^3 sphere at LC-39B. Recently, LNG has become a research focus of the CTL as well, as commercial launch providers such as SpaceX, United Launch Alliance (ULA), and Blue Origin are all using LNG as rocket fuel. LNG weathering is of particular interest, and testing is ongoing.

The Cryogenics Test Laboratory at KSC has a range of thermal insulation testing cryostats based on steady state boil-off calorimetry, using liquid nitrogen (LN_2) as a direct energy meter, in both cylindrical and flat-plate geometries (Figure 10), and comparative and absolute measurements. They are used for testing of foams, aerogels, plastics, powders, blankets, multilayer insulation, composites and panels according to ASTM C740 [16] and ASTM C1774 [17]. In principle, the cold side of the sample is maintained by liquid nitrogen at LN_2 temperatures while an electrical heater maintains a steady warm-side temperature from ambient up to 373 K. The steady boil-off flow rate of the liquid nitrogen provides a direct measure of the heat energy transferred through the thickness of the test specimen via the heat of vaporization. Nitrogen or other gases, including flammable fluids such as hydrogen, is supplied to the instrument vacuum chamber to establish a stable, moisture-free environment, with a controlled pressure range from 10^{-7} torr up to ambient pressure (760 torr).

The LN_2 cryostats cover a heat flux range from 0.1 to 400 W/m^2 with standard boundary temperatures 78 K and 293 K. The team also uses a set of commercial instruments for the measurement of thermal conductivity. The laboratory is equipped with a commercial Netzsch heat flow meter and C-Therm thermal conductivity analyser. They also use a wide range of cryocoolers, mainly from Cryomech (now part of Bluefors).

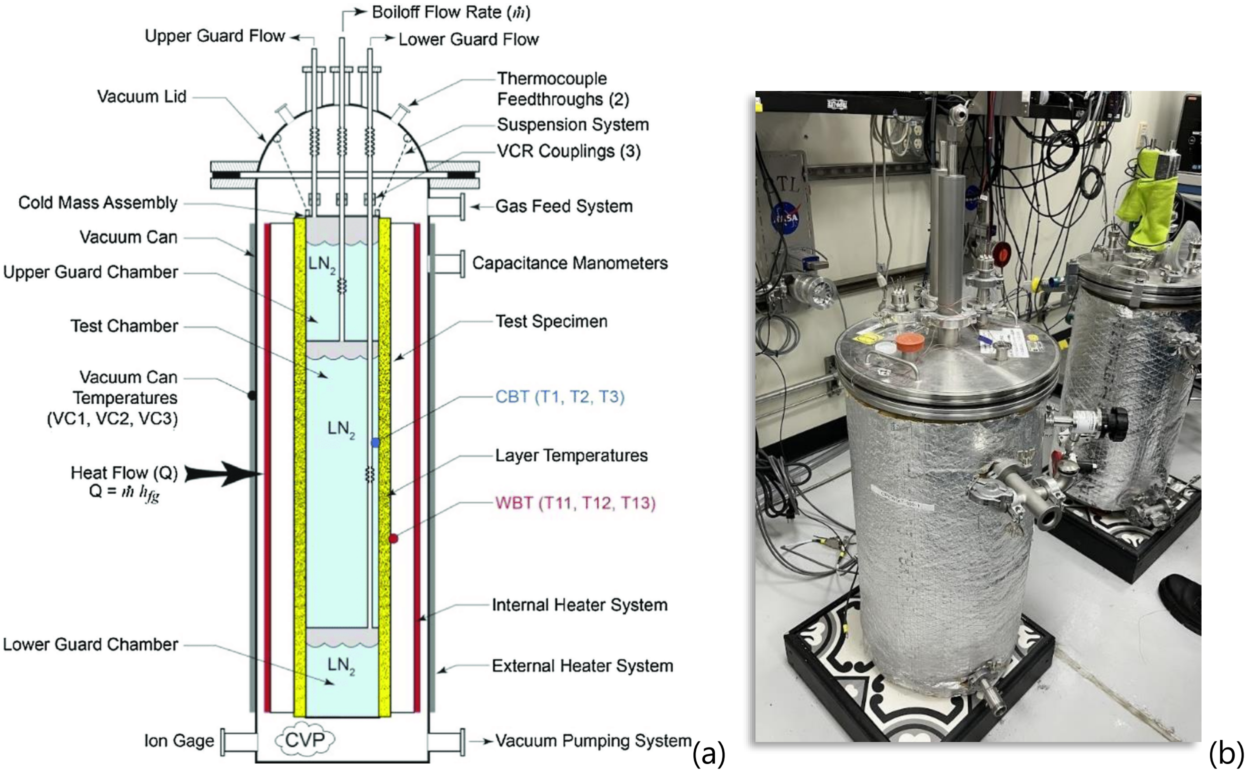


Figure 10 (a) Schematic of cylindrical boiloff calorimeter (Cryostat-100) and (b) Flat-plate (Cryostat-500) in the Cryogenics Testing Laboratory at NASA-KSC

In addition, the team has developed a Deep Space Irradiance Simulator (SIRS) system that mimics the solar-thermal environment of deep space. This comprises a high vacuum chamber that utilises a cryocooler and custom specimen enclosure to achieve background temperatures of roughly 15 K for a multi-day cooldown phase. When the sample and cold assembly reach steady state, a xenon lamp is switched on and the temperature is monitored while the sample warms, usually reaching maximum within about 4 hours, but can take anywhere from 3 to 12 hours depending on the emissivity. The test is terminated once the temperatures peak; the lamp and cryocooler are switched off and the system is allowed to warm back to ambient temperature under vacuum.

Key takeaways

The key takeaways of the fact-finding mission to the European and American laboratories can be summarised as follows:

Testing at cryogenic temperatures

1. The CryoMaK facility at Karlsruhe Institute of Technology (KIT) provides a one-stop-shop when it comes to testing advanced metallic alloys and composites at cryogenic temperatures, covering all the key properties (mechanical, thermal) and range of loading (mechanical).
2. The Mechanical Measurement Laboratory at CERN has considerable mechanical and thermal testing capability, with some overlap to that of CryoMaK's. There is significant effort invested in the development of fibre optic strain measurement using fibre Bragg gratings to measure strain and characterise coefficients of thermal expansion, which is particularly relevant in the high energy physics community.
3. The Electro-mechanical Testing Group at the National High Magnetic Field Laboratory (NHMFL) also houses a range of capabilities, more focused on materials relevant to high power magnets, but also possesses considerable expertise in the design and implementation of cryogenic measurement systems.
4. The Cryogenics Test Laboratory (CTL) at Kennedy Space Center focuses on research and development in thermal insulation systems and cryogenic component testing for both terrestrial and space applications.
5. Finally, the Materials and Processes Laboratory at NASA Marshall Space Flight Center (MSFC) offers testing at cryogenic temperatures at scale with approximately 25 electro-mechanical and servo-hydraulic test frames utilised for rudimentary test campaigns. A significant amount of work is being conducted on characterising the performance of thermal insulation foams.

Cryogenic testing in-situ liquid hydrogen

6. ET EnergieTechnologie are using a single wet closed system to perform mechanical testing in-situ liquid hydrogen with capabilities for both quasi-static and fatigue loading over a range of frequencies and loads.
7. HYPER Lab at Washington State University (WSU) performs liquid hydrogen research across multiple disciplines, focused on experimental testing and developing fluid dynamics and material models. Their CRyogenic Accelerated

Fatigue Testing system allows for small-scale in-situ hydrogen liquefaction and subsequent testing making this an ideal set-up for academic research.

8. NASA Marshall Space Flight Center (MSFC) have a unique set-up with extended capability, decades of experience and capacity of several test frames where testing in-situ liquid hydrogen is performed. These involve quasi-static, fatigue, and crack growth measurements for a range of materials intended for use in liquid hydrogen storage and distribution solutions.

In-situ gaseous hydrogen testing

9. The Mechanical and Hydrogen Testing Laboratory at PNNL has capability and expertise in mechanical testing of advanced materials at cryogenic temperatures, but also boosts an extensive suite of capabilities for investigating the effects of gaseous hydrogen on materials, incl. permeation, desorption, swelling, crack growth, fiction amongst others.
10. Sandia’s capability in evaluating the performance of metals and advanced alloys in-situ gaseous hydrogen (HPHT - high pressure high temperature) is unparalleled. This comes with extensive in-house expertise in design and implementation of complex experimental set-ups and associated health and safety systems.

It is vital that the UK develops measurement infrastructure to underpin hydrogen technology development by UK-based organisations and develop competence in support of supply chains with emerging energy and environment technologies. That should be inclusive of cryogenic and in-situ liquid hydrogen testing as well as in-situ gaseous hydrogen testing. More specifically:

- For cryogenic testing, there is an immediate need for both capability and capacity to cover the increasing demand for evaluation of mechanical and thermal properties of materials at 20 K.
- For in-situ liquid hydrogen testing, there is an immediate need for research-based testing facilities i.e., utilising small scale hydrogen liquification including in-situ, to boost materials research and innovation, followed by investment in large scale facilities like those at NASA Marshall Space Flight Center (MSFC).
- For in-situ gaseous hydrogen testing, the coordination of the existing facilities must be prioritised together with increase of current capability to higher pressures and temperatures like those at Sandia Laboratories.

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Appendix

Hydrogen projects

H2@Scale - is a U.S. Department of Energy (DOE) initiative that brings together stakeholders to advance affordable hydrogen production, transport, storage, and utilization to enable decarbonization and revenue opportunities across multiple sectors. More details at H2@Scale | Department of Energy

HydroGEN – is a DOE collaborative project that accelerates research, development, and deployment of advanced water splitting materials for clean, sustainable hydrogen production. HydroGEN makes unique, world-class national laboratory capabilities in photoelectrochemical, solar thermochemical, and low- and high-temperature electrolytic water splitting more accessible to academia and industry and supports national laboratory research in these areas. The water splitting is performed by heating up a metal oxide to 1000°C to drive off any oxygen and then the metal is exposed to water to produce hydrogen. The power to do this comes from solar renewable sources. More details at HydroGEN Advanced Water Splitting Materials Consortium | Department of Energy

HyMARC – The Hydrogen Materials Advanced Research Consortium (HyMARC) combines national laboratory expertise to develop clean, low-cost materials-based hydrogen storage systems that exceed the capabilities of physical storage (700 bar pressurized gas and liquid hydrogen) and meet U.S. Department of Energy (DOE) targets for stationery and transportation applications. In this way, HyMARC supports DOE's Energy Earthshots Initiative to provide clean, affordable hydrogen to achieve the nation’s climate and economic competitiveness goals.

HyMARC seeks to identify, develop, and optimize storage for hydrogen end uses with thus-far undefined technical targets, particularly for stationary applications. By employing a co-design strategy, systems modelling, and techno-economic analysis are directly coupled to materials discovery, design, and optimization to meet the requirements of specific use cases. More details at Hymarc

H2FIRST - Hydrogen Fuelling Infrastructure Research and Station Technology (H2FIRST) was a project launched by the U.S. Department of Energy’s (DOE’s) Fuel Cell Technologies Office (FCTO) within the Office of Energy Efficiency and Renewable Energy. The project sough to leverage capabilities at national laboratories to address the technology challenges related to hydrogen refuelling stations. The project was led by Sandia National Laboratories (SNL) and the National Renewable Energy Laboratory (NREL) and supported by a broad array of public and private partners.

The H2FIRST project was designed to ensure that fuel cell electric vehicle (FCEV) customers have a positive fuelling experience like conventional gasoline/diesel stations as vehicles are introduced (2015–2017) and transitioned to advanced fuelling technology beyond 2017. The H2FIRST activities were expected to positively impact the cost, reliability, safety, and consumer experience of FCEV stations.

HyBlend - The HyBlend initiative aims to address technical barriers to blending hydrogen in natural gas pipelines. Key aspects of HyBlend include materials compatibility R&D, technoeconomic analysis, and environmental life cycle analysis that will inform the development of publicly accessible tools that characterize the opportunities, costs, and risks of blending. HyBlend, consisting of over 30 partners and 6 national laboratories, ran a $15 million R&D project portfolio from 2021 to 2023. More details at HyBlend: Hydrogen Blending in Natural Gas Pipelines | Department of Energy

H-MAT - Working in collaboration with over 40 partners in industry and academia, the Hydrogen Materials Compatibility Consortium, or H-Mat, focuses on understanding the effects of hydrogen on the performance of polymers and metals used in hydrogen infrastructure and storage. H-Mat was launched in 2018 by the U.S. Department of Energy's Hydrogen and Fuel Cell Technologies Office in the Office of Energy Efficiency and Renewable Energy.

Applications currently being studied included hydrogen blending in natural gas pipelines, hydrogen storage vessels for fuelling stations and vehicles, hydrogen dispensing components (e.g., hoses, seals, nozzles), materials discovery, and accelerated test methods in hydrogen. Research activities include high-pressure testing of samples in hydrogen, multi-scale modelling of hydrogen effects, and microstructural engineering of materials to enhance resistance to hydrogen effects.

Consortium members include Sandia National Laboratories, Pacific Northwest National Laboratory, Oak Ridge National Laboratory, Savannah River National Laboratory, and Argonne National Laboratory. More details at H-Mat

SHASTA - The DOE’s Office of Fossil Energy and Carbon Management (FECM) is leveraging the unique capabilities and demonstrated expertise of four national laboratories — The National Energy Technology Laboratory (NETL), Pacific Northwest National Laboratory (PNNL), Lawrence Livermore National Laboratory (LLNL), and Sandia National Laboratories (SNL) — to determine the viability, safety, and reliability of storing pure hydrogen or hydrogen-natural gas blends in subsurface environments in a project named SHASTA or Subsurface Hydrogen Storage Assessment, and Technology Acceleration.

Hydrogen is emerging as a low-carbon fuel option for transportation, electricity generation, manufacturing applications, and clean energy technologies that will accelerate the United States’ transition to a low-carbon economy. However, a key challenge is to ensure the safe and effective storage of hydrogen. Large-scale storage of H_2 can be achieved by utilizing underground resources like how natural gas (NG) has been stored for the past century. Underground hydrogen storage (UHS) has the potential to provide the storage capacity required for the future hydrogen energy market. More details at Subsurface Hydrogen Storage | Department of Energy

eXtremeMAT – this project brings together seven U.S. DOE National Laboratories to harness the unique capabilities for materials design, high-performance computing, manufacturing, and characterization that exist across the U.S. DOE complex to accelerate the development of materials for service in extreme environments. The collaboration supports the U.S. DOE Office of Fossil Energy’s High-Performance Materials Program by addressing material integrity challenges inherent in highly efficient advanced energy systems. Specifically, this mission-focused team aims to improve both heat-resistant alloys and models for predicting long-term material performance. The consortium consists of the National Energy Technology and partner laboratories Ames Laboratory, Idaho National Laboratory, Lawrence Livermore National Laboratory, Los Alamos National Laboratory, Oak Ridge National Laboratory, and Pacific Northwest National Laboratory. The eXtremeMAT project is focussing on addressing hydrogen effects (eXtremeMAT-H2) on creep and embrittlement of metals as a function of microstructure. More details at eXtremeMAT

Three **Hydrogen Flagship Projects** represent an important contribution by Germany’s Federal Ministry of Education and Research (BMBF) to establishing the National Hydrogen Strategy (https://www.wasserstoff-leitprojekte.de/home).

H2Giga – To cover Germany's demand for green hydrogen, large capacities of efficient and cost-effective electrolysers are needed. Although efficient electrolysers are already on the market today, they are usually still produced by hand. The H2Giga flagship project will supports the series production of electrolysers.

H2Mare – At sea, the conditions are ideal for generating renewable electricity. The direct production of green hydrogen from offshore wind power facilities that are not connected to the grid can significantly reduce costs compared to onshore production. The H2Mare flagship project explores the offshore production of green hydrogen and other power-to-X products.

TransHyDe – Only a suitable transport infrastructure can ensure a successful hydrogen economy. Import, local distribution, and storage require different approaches. TransHyDe is further developing and testing technologies for the transport and storage of hydrogen. The aim is to find the right technology for each application.

Tools and resources

LAMMPS - this is a molecular modelling software using atomic potentials. There is also molecular dynamics modelling expertise in Albuquerque. LAMMPS is a classical molecular dynamics code with a focus on materials modelling. It is an acronym for Large-scale Atomic/Molecular Massively Parallel Simulator. LAMMPS has potentials for solid-state materials (metals, semiconductors) and soft matter (biomolecules, polymers) and coarse-grained or mesoscopic systems. It can be used to model atoms or, more generically, as a parallel particle simulator at the atomic, meso-, or continuum scale.

HELPR - is a modular, probabilistic fracture mechanics platform developed to assess the structural integrity of natural gas infrastructure for transmission and distribution of hydrogen natural gas blends. HELPR contains fatigue and fracture engineering models to allow fast computations while its probabilistic framework enables users to explore and characterize the sensitivity of predicted outcomes to uncertainties within the pipeline's structure and operation. HELPR development was supported by the Office of Energy Efficiency and Renewable Energy's (EERE) Hydrogen and Fuel Cell Technologies Office (HFTO) within the U.S. Department of Energy (DOE).

Hydrogen Plus Other Alternative Fuels Risk Assessment Models (HyRAM+) is a software toolkit that integrates publicly available data and models relevant to assessing the safety in the use, delivery, and storage infrastructure of hydrogen and other alternative fuels (i.e., methane and propane). The HyRAM+ risk assessment calculations incorporate probabilities of equipment failures for different components for both compressed gaseous and liquefied fuels, and probabilistic models for the effect of heat flux and overpressure on people. HyRAM+ also incorporates experimentally validated models of various aspects of release behaviour and flame physics. The HyRAM+ toolkit can be used to support multiple types of analysis, including code and standards development, safety basis development, facility safety planning, and stakeholder engagement. HyRAM+ was supported by the U.S. Department of Energy (DOE) Office of Energy Efficiency (EERE) Hydrogen and Fuel Cell Technologies Office (HFTO), the DOE EERE Vehicles Technologies Office (VTO), and the U.S. Department of Transportation (DOT) Pipeline and Hazardous Materials Safety Administration (PHMSA).

Data obtained through Sandia's experiments is made available to hydrogen and fuel cell technology stakeholders, and the general public, through the Technical Reference for

Hydrogen Compatibility of Materials and GRANTA MI-hosted Technical Database for Hydrogen Compatibility of Materials on-line resources.

Other facilities visited

Magnetic Resonance Laboratory at PNNL

Investigating catalysts and energy storage materials and how these work across a range of conditions so they can develop the design principles to create new ways to reduce emissions and remove carbon from the atmosphere.

Composite Materials Laboratory at PNNL

- The research group has also access to a comprehensive polymer composites laboratory equipped amongst other with:
- A wide range of composites preparation equipment incl. composite lay-up, filament winder, extrusion presses
- Evaporator being used to recycle carbon fibre using a catalytic hydrolysis process for epoxy resin at high temperature (240°C) and vapour pressure (~500 psi). The process is more environmentally friendly and releases fibres with up to 92% of original properties.
- Recycling thermoplastics and utilising monomers from the waste materials in spinning high strength fibre such as Kevlar.
- Plasma treatment for surface preparations particularly to enhance surfaces for lap shear strength and fracture toughness of adhesives.
- Thermoplastic forming capabilities.
- Extensive suite of analytical instruments to evaluate thermal property characteristics of wide range of materials.
- Ambient temperature nano-indentation
- Keyence VR-6000 series 3D optical profilometer

High Performance Materials Institute (HPMI) at Florida State University (hosted by Rebekah Sweat)

Summary of materials processing and characterisation facilities:

- 2 autoclaves for polymer composites processing
- A 10-ton graphite hot press, furnace up to 2,500°C.
- Carbon – carbon composites and ceramic composites, 3D ceramic printing
- Lay-up room > composite prep room > mechanical test laboratory > simulations > analytical characterisation laboratory
- Waterjet cutting facility.

- 5 kN mini dynamic load frame that can be used in an SEM.
- Low angle X-ray scattering, Raman spectroscopy and mapping, AFM, contact angle measurement, AFM + IR,
- Thermo Scientific Helios G4 UC with EBSD, EDS and FIB - cryo-stage and micro-stage capabilities.
- Simultaneous Thermal Analyser – DSC & TGA up to 2,400°C
- ARES-G2 Rheometer and orthogonal DMA down to -100°C (TA Instruments)

The College of Engineering at FSU, within which the HPMI is located, are exploring opportunities for LH_2 testing.

Funding for the High-Performance Materials Institute (HPMI) within the Florida State University (FSU) is mainly from the Department of Defence (DoD).

National High Field Magnet Facility at NHMFL (hosted by Jun Lu)

The facility at the Florida State University hosts a series of world-record magnets that can be used by external organisations to conduct measurement techniques across condensed matter physics, materials research, magnet engineering, chemistry, biochemistry, geochemistry, bioengineering, and biology. The magnets are used to explore the behaviour of exotic metals, quantum inclusions etc, measuring thermal conductivity, specific heat capacity etc.

Seven distinct user facilities offer unique capabilities in high magnetic field research, covering:

- Advanced Magnetic Resonance Imaging and Spectroscopy (AMRIS) Facility
- DC Field Facility
- Electron Magnetic Resonance (EMR) Facility
- High B/T Facility
- Ion Cyclotron Resonance (ICR) Facility
- Nuclear Magnetic Resonance/Magnetic Resonance Imaging (NMR/MRI) Facility
- Pulsed Field Facility

Proposals can be submitted to use the facilities free of charge. Successful applicants are granted one week of magnet time (8 hrs/day), 200 MWh of energy and 250 litres of LHe.

Key contacts

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