

Effect of Cryogenic Thermal Cycling on the Response of a Fiber Bragg Grating Sensor

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ABSTRACT

The manufacturing and use of composites hydrogen tanks for transport applications faces a series of very important challenges among which one of the most critical is the definition of a structural monitoring system that is able to adapt to the conditions present in the tank.

Among the different ways of storing H₂, two of the most interesting from the point of view of gravimetric and volumetric density are the storage of H₂ in liquid form LH₂ or in the form of cryo-compressed gas CcH₂ in both cases the tank structure (either the liner for class III and IV or the composite structure itself for class V tanks) must be able to withstand very important thermal variations in temperature during the processes of filling and emptying the tank, i.e. able to withstand high thermal cycling requirements. The research work carried out has focused on studying the impact of such thermal cycling on the sensor response. For this purpose, FBG sensors have been integrated in several coupons that have been subjected to multiple thermal cycling (20°K-80°K). After each thermal cycling, a reference tensile test has been performed on the coupons, recording the responses of the sensors to see the effect of the accumulation of the cycling, in parallel after each cycling, inspections have been performed by micro computed tomography to try to check if the possible cause of the variation in the response is associated with micro cracking in coupon-sensor interface.

The final objective of the research is to ensure the reliability of the response of the FBG sensors for H₂ SHM application.

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INTRODUCTION

The European Union, as part of its strategic plan known as the Green Deal, has set highly ambitious targets for reducing greenhouse gas emissions in the transportation sector (land, sea, and air). These goals include a 55% reduction by 2030 compared to 1990 levels and a 90% reduction by 2050 (Fetting, 2020) [1].

The Green Deal, in addition to setting targets for transforming how we move, promotes a focus on technological development centred on green technologies across Europe. It encourages industries directly or indirectly connected to transportation in investing in green innovation and development.

Among the various options to achieve the transportation sector's emission reduction target, the use of hydrogen (H₂) as a fuel, has emerged as a particularly promising alternative to biofuels. The use of H₂ as an energy carrier offers multiple advantages, such as:

- It can be stored in a relatively simple way, either as pressurized gas or liquid, and transported similarly to natural gas, taking advantage of the existing infrastructure.
- It helps to integrate renewable energy into the energy system, by storing and transporting the excess electricity production (green hydrogen).
- It can be compressed, liquefied, or converted into other fuels due to its high energy density and low physical density, allowing for better energy utilization.

In the transport sector, multiple projects have been developed to implement H₂ as an energy carrier. In the aeronautical sector, Airbus is developing the Zero-e project to study different aircraft designs that use H₂ as fuel, either for direct combustion or for use in fuel cells (Airbus, 2025) [2]. In the railway sector, both Talgo and CAF are developing hydrogen train projects such as the Talgo-Repsol project and the FCH₂Rail project by CAF (FCH₂RAIL, 2025) [3], among others. In the maritime sector, manufacturers like Navantia have made significant investments in hydrogen, with the strategic development of the S-80 class submarines, where hydrogen will provide the necessary energy for long dives. In the automotive sector, H₂ has been present for many years, and it is now possible to find hydrogen-powered vehicles on the market, such as the Hyundai Nexo and the Toyota Mirai.

Hydrogen can be stored dissolved in other substances such as ammonia or methanol, in compressed gaseous form, or in liquid form at cryogenic temperatures. Storing it dissolved is an alternative that offers a high gravimetric storage capacity but requires the use of devices such as reformers to extract the H₂ from methanol or ammonia. This means that for aerospace, terrestrial, and in many naval applications direct storage of H₂ is preferred to avoid complex systems.

The aviation sector requires the gravimetric index of fuel tanks to be as high as possible to minimise weight to the greatest extent. As a result, the technologies currently under development are increasingly focused on the use of liquid hydrogen (LH₂).

Despite offering higher density and storage efficiency, the process of liquefying hydrogen at -253°C is both energy- and time-intensive. It is estimated that around 40% of the energy content is lost during this process, compared to just 10% for other storage methods, such as compressed hydrogen (Barthelemy et al., 2017) [4]. Additionally, liquid hydrogen is challenging to store over extended periods due to product loss through evaporation.

To effectively manage storage at $-253\text{ }^{\circ}\text{C}$, high-efficiency (vacuum) insulated vessels are required. These vessels typically consist of an inner pressure vessel and an external protective jacket. To minimize thermal conductivity between the inner vessel and the outer jacket, insulating materials such as perlite (in powdered form) or super insulation (layers of aluminum film) are employed.

An alternative is Cryo-compressed storage, which combines the properties of both compressed gaseous hydrogen and liquefied hydrogen storage systems. It is designed to minimize boil-off losses (dormancy) from liquefied hydrogen storage while maintaining a higher system energy density. Hydrogen is stored in an insulated tank that can withstand cryogenic temperatures (20 K) and high pressures (at least 30 MPa) at ambient temperature. The tank's ability to endure high pressures allows for greater pressure increases before hydrogen needs to be boiled off. These cryogenic pressure vessels significantly extend the time before evaporative losses occur during operation, thus increasing storage autonomy.

As an example, the BMW Group has started validation of cryo-compressed hydrogen storage for hydrogen vehicles with high energy and long-range requirements (BMW, 2014). The diagram depicted in Figure 3.1 reported by BMW [5] shows that cryo-compressed H₂ enables high storage density (80 g/l). Being the cryogenic gas is denser than liquid hydrogen.

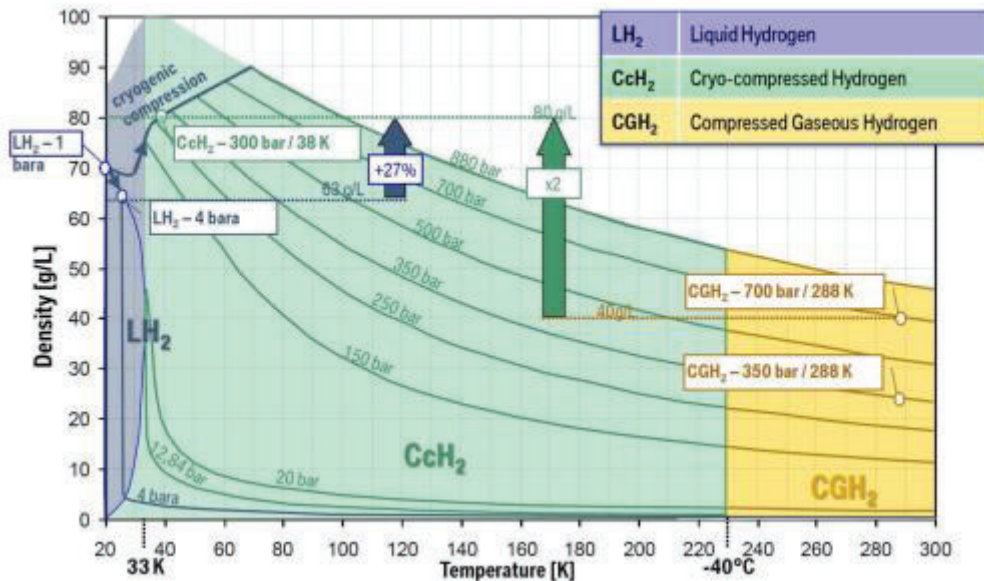


Figure 1: Hydrogen density versus pressure and temperature from BMW (BMW, 2014)

Cryo-compressed storage tanks can be filled with hydrogen at any state between 20 K liquid H₂ and ambient temperature gaseous H₂. Filling the tank with compressed gas instead of liquefied hydrogen is expected to be more economical. In terms of infrastructure, cryo-compressed tanks offer refuelling flexibility as they are compatible for gaseous and liquid.

Design of Cryo-compressed hydrogen tanks: The architectures of pressure gas storage tanks available can be observed in Figure 3.2. The tanks are classified into five categories:

- Type I tanks, made entirely of metallic material.
- Type II tanks, which include a metallic liner and partial reinforcement in composite.
- Type III tanks, which combine a metallic liner with full reinforcement in composite.
- Type IV tanks, which use a polymeric liner and reinforcement in composite.
- Type V tanks, made entirely of composite material.

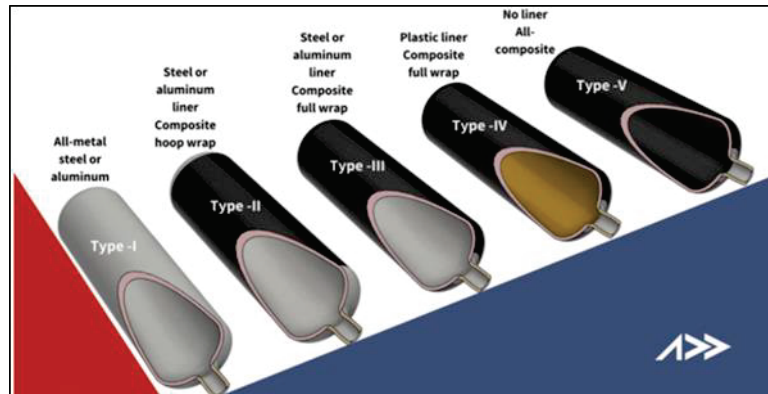


Figure 2: Different architectures of pressure gas storage tanks (source: ADD composites)

Type V tanks offer significant advantages over the others, primarily due to their weight reduction, which can reach a 25% compared to Type I tanks (Air et al., 2023) [6]. Additionally, the absence of a liner eliminates thermomechanical issues at the liner-reinforcement interface, caused by differences in the coefficients of thermal expansion (CTEs) of the materials. This aspect is particularly critical in Cryo-compressed (CcH₂) applications, where tanks are subjected to high mechanical stresses due to storage pressure and significant temperature variations during filling and emptying cycles.

The high design and manufacturing costs have driven the aerospace and aeronautical sectors to adopt tanks with longer life cycles. In the aerospace sector, there is a shift from disposable tanks, used in single or multi-stage rockets with a mission-limited lifespan, to reusable tanks, such as those used in the Falcon 9 or Starship, whose service life can extend to several years. In aircraft transport, current fuel tanks have a service life of 20 to 30 (with routine inspection), so it is expected that hydrogen technology will reach these standards. This approach implies that tank design must consider both functional and structural aspects throughout its entire life.

Among the most critical functional aspects is the tank's permeability to potential leaks. Load conditions below ultimate values can cause matrix cracking in the composite, creating channels through which hydrogen can escape more easily. From a mechanical perspective, the walls of the tank are subjected to biaxial loading during operation, and the filling and emptying cycles expose the material to thermomechanical cycles, with temperatures ranging from room temperature (RT) to cryogenic conditions, and pressures varying from atmospheric pressure to 350 bar.

To address these challenges, experimental methodologies are required to determine the material properties and tank permeability under service conditions. Additionally, inspection tools are needed to perform Structural Health Monitoring (SHM).

MATERIALS AND METHODS

The fibers were Polyimide-coated, provided by FISENS Technologies, and bonded with different adhesives (EA2A adhesive from Tokyo Measuring Instruments, X60 Adhesive from HBM and NOA 61 Adhesive from Norland products). The optical interrogator used for the testing was LUNA Si155. The coupon composite material was a thin ply from TeXtreme composites.

The development of this experimental work is being structured in 3 different phases that try to study the feasibility of using fiber optic sensors in cryogenic conditions:

- Phase 1 (FBGS stand-alone testing) consists of studying the variation in the behavior of the free FBGS sensor, for this purpose INTA has developed a tool (figure 4) that allows thermal cycling (1200 cycles 20°K-77°K) of fiber optic sensors by immersion in a dewar tank of liquid helium, using this technique FBGS sensors have been cycled up to 12000 cycles.
- Phase 2 (Adhesive testing) consists of studying the behavior of the interface between the FBGS sensor and the composite material substrate, for this purpose different adhesive bonds have been thermally cycled (1200 cycles 20°K-77°K) to determine the resistance capacity of the different adhesives. To perform the cycling, a resistive heated sample holder (figure 5) specifically designed for the project and the Oxford cryostat model 100KN, S/N 40786 (figure 6) available at INTA have been used.
- Phase 3 (FBGS-Adhesive Assembly Testing) consists of studying the behavior of the FBGS-adhesive sensor assembly, for this purpose FBGS sensors have been integrated in ASTM-D-3039 tensile specimens which have been cycled (1200 cycles 20°K-77°K) using again the heated specimen holder and oxford cryostat. Once cycled, a reference tensile test (defined deformation level) is performed on the specimens and the results are compared.

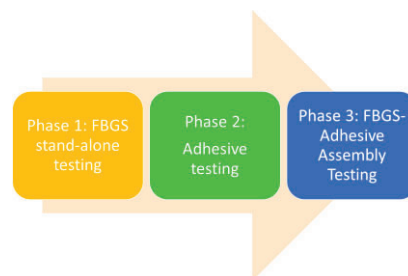


Figure 3: Experimental Work phases.



Figure 4: FBGS stand-alone thermal cycling tool.

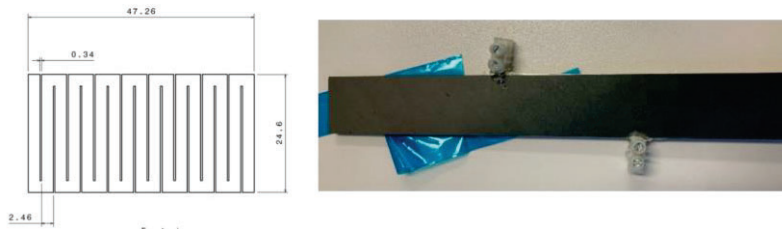


Figure 5: INTA's resistive heated sample holder.

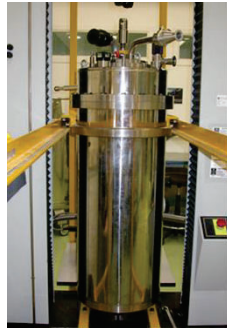


Figure 6: Oxford cryostat model 100KN, S/N 40786

The thermal cycling characteristics (maximum and minimum temperatures) have been controlled by means of the immersion and air exposure time in the case of phase 1 or by means of the current intensity in phases 2 and 3, achieving high levels of repeatability in all phases.

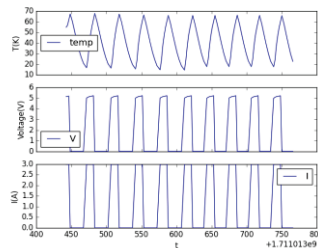


Figure 7: Example of temperature profile during phase 2 and phase 3.

At the date of writing this paper, partial data are available for phases 2 and 3, as 600 of the 1200 cycles planned have been carried out; therefore, only partial results will be presented in the results section.

RESULTS

The results of phase 1 shows that there is a weak influence over the stand alone FBG sensor response, in the following (Figure 8), partial results can be observed. The graphs

show the temperature of the sensors on the left hand axis and the response of the cycled sensors on the right hand axis.

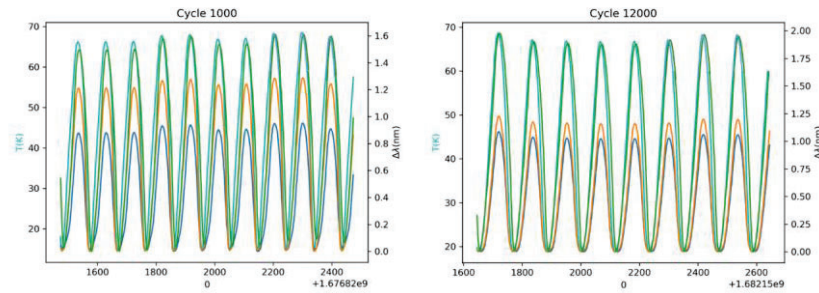


Figure 8: Sensor Response after 1000 and 12000 cycles.

The results of phase 2 can be observed in the following table (table 1) where we can observe that only the adhesives EA2A adhesive from Tokyo Measuring Instruments, X60 Adhesive from HBM has withstood the 600 cycles correctly without showing any apparent debonding, the latter having been verified by non-destructive μ CT inspections.

| Adhesive | 300 Cycles (20°K-77°K) | 600 Cycles (20°K-77°K) | 900 Cycles (20°K-77°K) | 1200 Cycles (20°K-77°K) |
|----------|------------------------|------------------------|------------------------|-------------------------|
| EA2A | Go | Go | | |
| X60 | Go | Go | | |
| NOA61 | No go | | | |

Table 1: Different adhesives failures after thermal cycles.

The results of phase 3 are still to be completed as for phase 2, so far 4 different specimens have been cycled with polyimide sensors (300 cycles of the 1200 planned) integrated with the adhesives that showed the most promise in phase 2 (EA2A and X60), after each cycling a reference tensile test was performed on them and a tomography (Figure 9) and a recording of the spectrum was carried out. The objective after the 1200 cycles is to observe the possible relationship between the occurrence of micro-cracking due to thermal cycling, the sensor-coupon adhesive debonding and the response of the sensor.

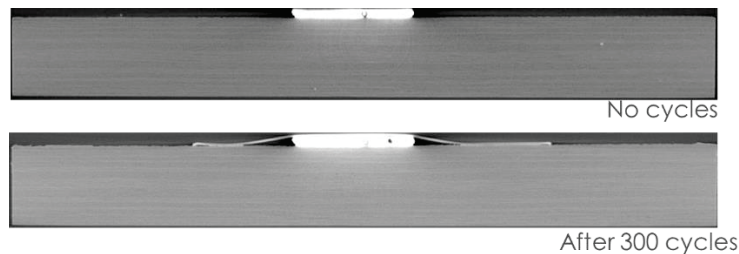


Figure 9: Example of μ CT phase 3 of FBGS interface.

CONCLUSIONS

The development of safer and more efficient hydrogen tanks requires them to be equipped with structural health monitoring (SHM) systems that can predict the appearance of damage or leaks, or provide data to digital twins. A fundamental aspect of SHM systems is the sensors; in the case of liquid hydrogen (LH2) or cryogenically cooled (CCH2) tanks, it is important to note that not all sensors are capable of working under the cryogenic conditions present. The present work aims to evaluate the feasibility of its use through various experiments.

Based on the results available so far, FBGS sensors represent a very interesting alternative for this use due to their stability in response to the thermomechanical conditions present, including cycling, and the ease with which they can be integrated.

ACKNOWLEDGMENT

The work described in the paper has been financially supported by the “Agencia Estatal de Investigación” through project CRYFTO (PID2021-127879OA-C22)

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