High-Store – HYS2172

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Contents

1   Glossary

Acronym	Meaning
Bar	A unit of pressure defined as 100 kilopascals
Barg	A unit of gauge pressure, i.e. pressure in bars above ambient or atmospheric pressure
CapEx	Capital expenditure
cDAQ	Data acquisition device
CO_2	Carbon dioxide
CSC	Chesterfield Special Cylinders Ltd
Dehydrogenated	Extracting hydrogen
H_2	Hydrogen gas
Hydrogenated	Adding hydrogen in
IP	Intellectual Property
J4IC	Joining 4.0 Innovation Centre (Lancaster University)
kg	Kilogram
MEGC	Multiple element gas container
Mg	Magnesium
Mt	Mega tonnes
OpEx	Operational expenditure
PCI	Pressure Composition Isotherm
SAM	Site availability model
TWI	TWI Ltd
UoN	University of Nottingham
WP	Work Package
Wt.%	Weight Percent

2   Executive summary
2.1   Introduction
The products being developed within High-Store address high volume hydrogen storage/supply in an extremely safe form (chemically captured solid) compared to current compressed gas alternatives.
2.2   Need/Problem
The consumption of green hydrogen as a fuel is increasing as the supply, and production by electrolysers' increases. Currently gas compression is used to pressurise gas cylinders in a range 350barg to 700barg, which uses a high amount of energy (which can be up to 20% of the calorific value of hydrogen) to compress the gas. The mechanical compressor itself also has high CapEx and OpEx cost. As hydrogen's popularity increases, it creates a scenario where the road network experiences increased levels of lorries transporting hydrogen. The burden to drive down delivery costs results in carriage at increasingly higher pressures; with a serious consequence of rising safety concerns and the energy used for this compression.
2.3   High-Store
High-Store is a technology that stores hydrogen at low pressure but stores as much gas as though it were at very high pressure. The gas is stored chemically as a metal hydride, therefore, the hydrogen is converted to a solid form with a modest overpressure, up to 30bar, which is safer for transportation and bulk storage.
2.4   Technology development
The technology is based on postdoctoral research and the BEIS funded research to build a hydrogenation/dehydrogenation demonstration system at Nottingham University. The funding has developed the test vessel and internal architecture and the gas delivery and flow measurement equipment.
2.5   Conclusions
The challenge was to design and build a metal hydride store that has high capacity, is cost effective and comprises innovative internal architecture in order to heat the store, with good kinetics. This involved multiphysics modelling to model the reaction from the magnesium bed and to manage the heat and optimise the maximum capacity to be released. The model predicts kinetics and temperatures in the bed for different designs and informed the test prototype design. This is a significant piece of work for a 9-month period. All steps have been completed. The process has been physically tested, thermal cycling being undertaken, achieving 2.17 wt.% storage, which is equivalent to a volumetric capacity of $0.014kg/lH_2$. The target was 4.17wt.% $0.026kg/lH_2$. Whereas liquefied H_2 would be $0.071 kg/lH_2$. A primary objective of phase2 would be to improve the High-Store capacity towards the maximum of 6.5wt% which is a volumetric capacity of $0.041kg/lH_2$. While the storage of the hydrogen is a low energy process, the recovery (dehydrogenation) to reform the hydrogen in to gas takes more energy. It has been calculated this would be approximately a third of the energy contained within the hydrogen.

3   Overview of Project

The products being developed within High-Store are key components of the emerging hydrogen economy. The specific needs addressed are for high volume hydrogen storage/supply in an extremely safe form compared to current alternatives. The project addresses the storage vessel with the gas delivery and electrical heating system, it does not include equipment for hydrogen generation or associated functions. The key performance indicators address:

- The delivery of a metal hydride dehydrogenation technology offering the equivalent volumetric storage capacity of liquefied hydrogen, but avoids compression or liquefaction penalties.
- A design that creates Hydrogen from electrolyser technologies at 30 bar, being used to refuel the storage vessel at 30 bar, but containing a similar volume of hydrogen as if it were liquefied.

3.1   Objectives

- Utilise magnesium as a low cost metal hydride to store and transport hydrogen at low pressures (less than 30barg) utilising thinner wall less expensive pressure vessels.
- Investigate the potential of an internal tank structure with internal electrical heating elements (rather than more expensive shell and tube heat transfer fluid type approaches) to release the hydrogen during the hydrogenation and dehydrogenation stage.
- Model the reaction front kinetics using a multiphysics approach, in order to determine the optimum configuration of heater elements and fins to hydride.
- Explore and model temperature ranges to maintain temperature envelopes of vessel and design brief.
- Design bespoke heating and fin configuration that can be inserted into a pressure vessel. Build a prototype to confirm the simulation model, test initial design and optimise positioning of heaters and fins. Demonstrate this approach is technically feasible in supporting hydrogen supply.

3.2   Description of Technology

Established hydrogen storage technologies involve either mechanical compression or liquefaction. The most common form of hydrogen storage is to mechanically compress hydrogen gas to pressures between 100 and 700 bar depending on the application. This has advantages as compressing gas to higher pressures increases the volumetric efficiency. Compressing hydrogen gas has several disadvantages. Energy is required to achieve compression e.g., compression of hydrogen gas to 700 bar uses approximately 6.0 kWh/kg, leading to approximately 1.3kg of CO_2/kg. Capital investment and maintenance

costs are incurred for the use of mechanical compressors. Specialist vessels are required for gases at high pressure. Pressurised gas containment can pose several safety issues.

Hydrogen liquefaction is also advantageous as it provides high volumetric efficiency superior or similar to that achieved by high pressure compression. Hydrogen liquefaction also possesses several disadvantageous, such as energy is required to reduce the temperature of the hydrogen to -253°C, liquid hydrogen can burn or explode if leaked, and specialist vessel and pipe networks are required to store or transfer it.

The High-Store concept utilises the high storage capacity of Mg hydride. Mg powder is contained in a pressure vessel rated for modest working pressure of 40bar. Mg powder will react with hydrogen if it is heated to above 340°C and supplied at a pressure greater than 8barg. Once the hydrogen has reacted with the Mg powder it is chemically bound in the metal particles and stored as a solid. If the powder and the pressure vessel are allowed to cool then the hydrogen will remain in solid form i.e., as a metal hydride. To release the hydrogen the temperature of the store heat must be raised beyond 340°C. The higher the temperature the higher the pressure at which hydrogen is released from the metal. The reaction between Mg and hydrogen is highly reversible enabling the process to be repeated as required. The challenge is to provide a practical heating system for a pressure vessel which efficiently delivers the required thermal input to facilitate the reaction between Mg powder and hydrogen. The relatively low thermal conductivity of the Mg powder necessitates the provision of thermal management to ensure that heat can readily penetrate uniformly through the powder bed. The High-Store concept will comprise of a pressure vessel containing Mg powder. Embedded in the Mg powder are electrical heaters the position of which is optimised for heat distribution. In addition to the heater extended surfaces will be used to promote heat transfer. The heaters will be connected to an electrical power source located externally to the pressure vessel.

3.3　　Storage Medium Selection

The selection of magnesium as a hydrogen storage medium for this application and not other technologies or hydride alloys is based on the following:
1) Magnesium is the planet's 7th most abundant material and is already mined and processed on an industrial scale i.e., a supply chain is already established for large scale industrial applications.
2) Due to the abundance of magnesium it has the potential to reach a low cost point at industrial scale of use. Current 'large academic/research' volume pricing for magnesium powder is around £1 per kg varying in time with market forces (September 22). Energy cost changes in recent months, whether provided by

electricity, or fuels (diesel or petrol etc.) may be a critical limiting factor for the longer term magnesium pricing. Calculations will assume a price of £4/kg to offset likely fluctuation and rises.

3) The storage capacity of magnesium is 7.6wt.% of hydrogen. That is 100kg of MgH_2 stores up to 7.6kg of hydrogen. In reality research has shown the usable capacity for Mg type stores is closer to 6.0 to 6.5wt.% hydrogen when cycled.

High-Store overcomes the challenges of counterfactual technologies as follows:

1. Intrinsic safety advantages in the event of a line breach, i.e. comparatively low pressure storage less than 30barg compared with current transportable storage solutions (>230barg, up to 500barg). High-Store also has a very low free volume of gas to escape in the event of a leak or rupture.

2. Low capital and operating costs. Due to the Mg kinetics High-Store is able to meet the 30barg refuelling requirement and by-passes any compression stages used by compressed hydrogen gas storage. This means reduced ownership costs, removing complexity and cost of compression, and energy usage which could potentially be offset by High-Store's higher electricity/heat usage which drives dehydrogenation (i.e. release of hydrogen from the metal matrix). The low storage pressure also enables thinner walled vessels to be used for a given volume compared to compressed gas pressure vessels, reducing supply and transport costs.

3.4   Description of High-Store Prototype

A storage vessel is required to store hydrogen at low pressure nominally 30barg. While the principal storage mechanism is to capture hydrogen as a hydride and therefore is not necessarily under pressure, but due to a free space volume inside the vessel, which is pressurised by gaseous hydrogen from an external source, a small volume of free hydrogen remains in the vessel at 30barg even after the hydride is fully formed. Therefore this free volume can be liberated/released, whereas the hydride component cannot, until electrically stimulated. When the system is electrically powered a range of pressures could be experienced. For example up to 30barg when receiving hydrogen from a source such as an electrolyser, and a likely delivery target of 5barg to feed a fuel cell.

The vessel's head-space serves an important role to accommodate expansion of the hydride on formation. Much in the same way as water expands into ice, so the metal will expand in to the metal hydride, the head-space allows for this safe expansion to naturally occur. The dehydrogenation circuit is used to effect the bidirectional transport of hydrogen (charging or discharging) depending on the hydrogen pressures employed and the temperature the matrix is heated to. The dehydrogenation circuit comprises three principal systems.

1.   Heating and distribution.
2.   Wiring.
3.   Gas take-off.

The project has taken a few different pathways in arriving at the final version. This has been due to the technology challenges encountered during the development process and the learning gathered. The changes in direction have resulted in extra efforts in cylinder technologies, initially these focused on a wide mouth cylinder, which made introduction of internal components easy, however with production line failure, the project had to consider alternatives with a small cylinder mouth. In each case detailed multiphysics modelling occurred that modelled the reaction fronts and thermal conditions throughout the store with different heating configurations and whether under hydrogenation or dehydrogenation of the store.

The small entrance size presented a challenge in placing the many components internally. Because the heaters are small, having a low surface area, and being required to work at a high power density but within (effectively) an insulator, gave a very complex series of requirements to overcome. Detailed simulations were undertaken establishing the likely power requirement and heating arrangements, which were all required to fit through an 18mm diameter vessel port.

3.4.1   Explanation Endo/exo thermic reactions

The rate of reaction of a metal hydride is a function of pressure and temperature. Under hydrogenation, the storage media reacts with hydrogen and heat is released (exothermic), however in this case the media is not cooled, the heat being lost to the vessel.

Under Dehydrogenation the hydrogen that has previously reacted with the media is released, by supplying heat (endothermic).

The rate at which hydrogen can be supplied and released from a metal hydride store is dependent on the reaction rate. The reaction rate is strongly dependent upon the reaction temperature and the pressure of the hydrogen gas.

3.4.1.1 Equilibrium Pressure

For any given temperature there is an equilibrium pressure for the reaction. If the pressure of the hydrogen is above the equilibrium pressure associated with the temperature of the media bed, then conditions favour hydrogenation and hydrogen gas will react with the powder – hydrogenation. If the pressure of the hydrogen is below the equilibrium pressure associated with the powder

temperature, then conditions favour dehydrogenation. Hydrogen gas will be released from the metal – dehydrogenation, as indicated by the schematic Figure 1. In reality there is a hysteresis between hydrogenation and dehydrogenation and the modelling design took that into account.
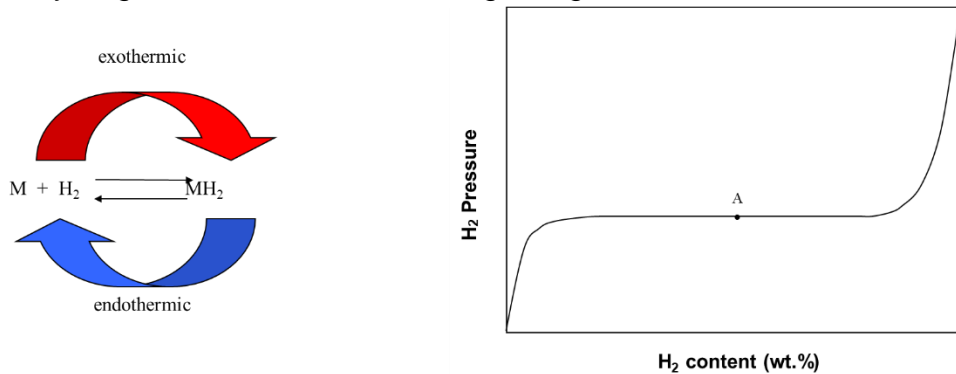

Figure 1 Idealised PCI (Pressure Composition Isotherm), showing the effect of pressure on the amount of hydrogen stored in a metal hydride at a constant temperature. Point A denotes the plateau pressure, which will be higher at higher temperatures.

3.4.2   Heat Transfer

During hydrogenation heat is released from the powder. Unless heat is transferred out of the powder bed, then the temperature of the powder bed will rise. This in turn raises the equilibrium pressure which results in slowing the reaction and ultimately halting the reaction altogether i.e., no more hydrogen will be stored in the media.

During dehydrogenation hydrogen is released from the powder whilst heat is transferred into the store. If the transfer of heat is insufficient then the temperature of the powder bed will decrease which will lower the equilibrium pressure. As the equilibrium pressure falls then the rate of dehydrogenation will decrease and will ultimately stop altogether i.e., no hydrogen will be released.

For High-Store it is crucial that the bed temperature is raised to at least 360°C otherwise the reaction rate of magnesium is too slow for practical operation. The reaction rate is a function of temperature. In this respect heat management of the powder bed is critical to the rate at which hydrogen can be transferred in and out of the vessel.

3.4.3   Model Domain and Description of SAM

The complex interdependency that exists between temperature, pressure, heat transfer and reaction rate requires a numerical model to predict the performance of the High-Store. The kinetic reaction for magnesium is further complicated by the physical mechanisms that occur as hydrogen permeates through each

individual metal particle. In the case of magnesium these mechanisms are difficult to represent using standard rate equations so a more sophisticated model has been derived. The site availability model (SAM) developed at UoN provides a greater reliability when simulating the performance of magnesium behaviour under different temperature and pressure scenarios, shown in Figure 2.
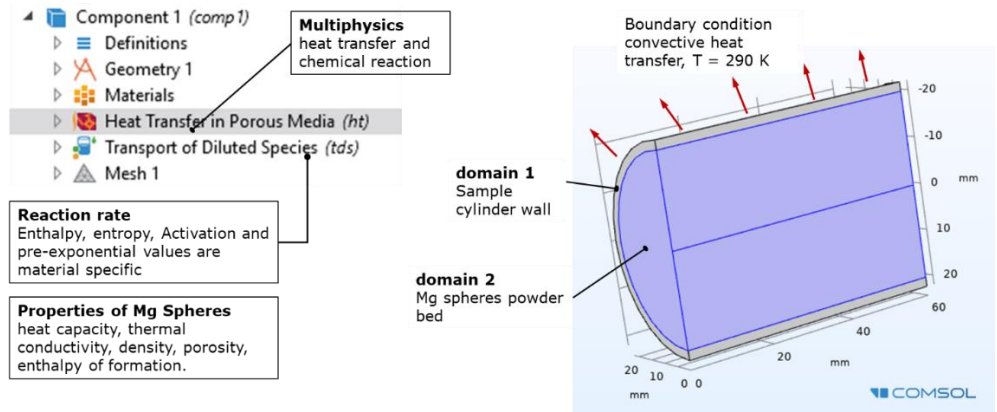

Figure 2 The SAM model.

4   Experimental/modelling results and conclusions

4.1   Experimental Objectives

- Model the store and thermal characteristics.
- Assemble, fill and commission prototype store.
- Activate store and establish consistent hydrogen capacity.
- Demonstrate successive hydrogenation and dehydrogenation cycles.

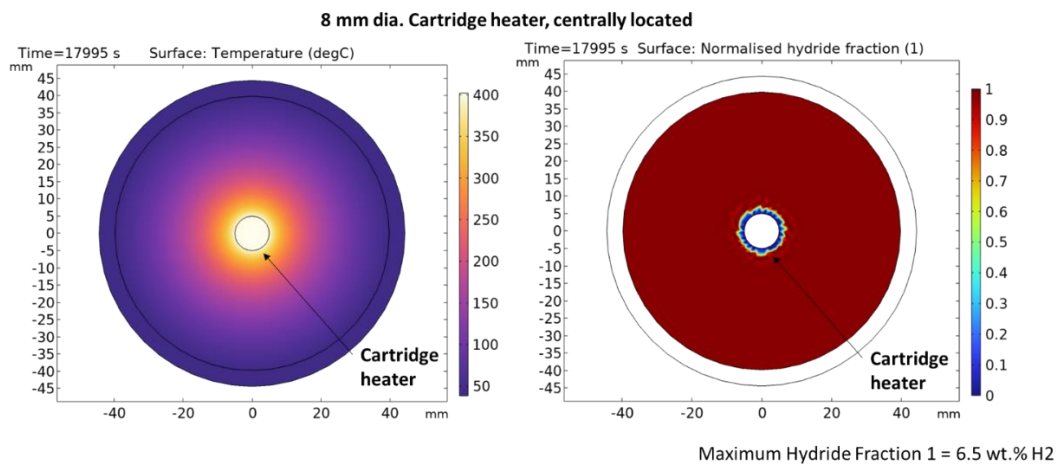### 4.2   High-Store Thermal Analysis

Figure 3 Single 8mm cartridge heater.

The poor thermal conductivity of the bed means that the time it takes for heat to transfer from the heater location through the powder bed is significant.

Dehydrogenation requires the supply of heat which means a single heater located centrally in the store is unable to distribute heat through the store and this limits the amount of hydrogen that can be released. As the number of heaters is increased this promotes heat transfer and improves the rate of dehydrogenation. Figure 3 shows a section through an 8mm heater and media, the left hand picture shows the hot zone, the right hand picture shows the amount of conversion in blue.

Figure 4 on the 'left hand side' shows the temperature by distance from the centre. It can be seen that the temperature drops off quickly even 10mm from the centre. The 'right hand side' shows by simulation the effective conversion of hydride being 100% hydride at the top i.e. no conversion, and full conversion at the bottom. Therefore in this case very little has been converted.
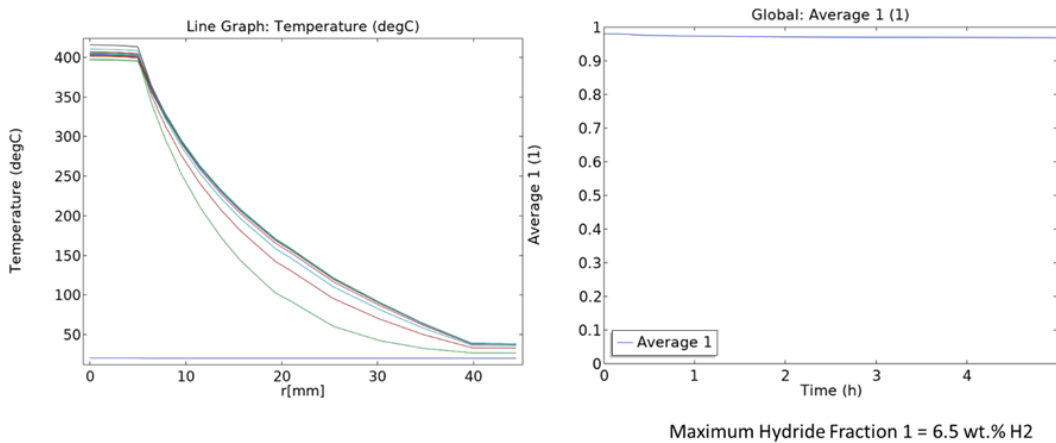

Maximum Hydride Fraction 1 = 6.5 wt.% H2

Figure 4 Heater profile from the centre.

In the case shown in Figure 5, the heaters have been increased to 3, the area in the centre of the heaters benefits from an improved conversion rate. However externally the conversion is extremely low. Figure 6 shows the percent conversion of the hydride when using four and five heaters, the percentage conversion improves with each addition.
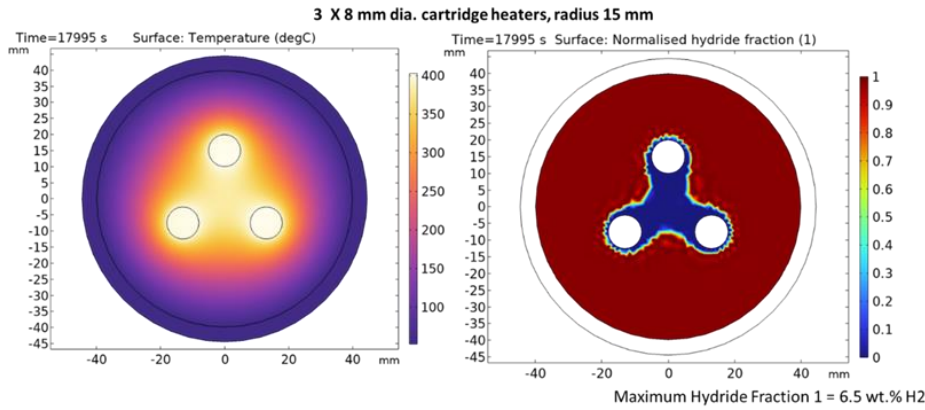
3 X 8 mm dia. cartridge heaters, radius 15 mm

Time=17995 s   Surface: Temperature (degC)

Time=17995 s   Surface: Normalised hydride fraction (1)

Maximum Hydride Fraction 1 = 6.5 wt.% H2

Figure 5 Section through 3 heaters and media.

4 X 8 mm dia. cartridge heaters

Global: Average 1 (1)

5 X 8 mm dia. cartridge heaters

Global: Average 1 (1)

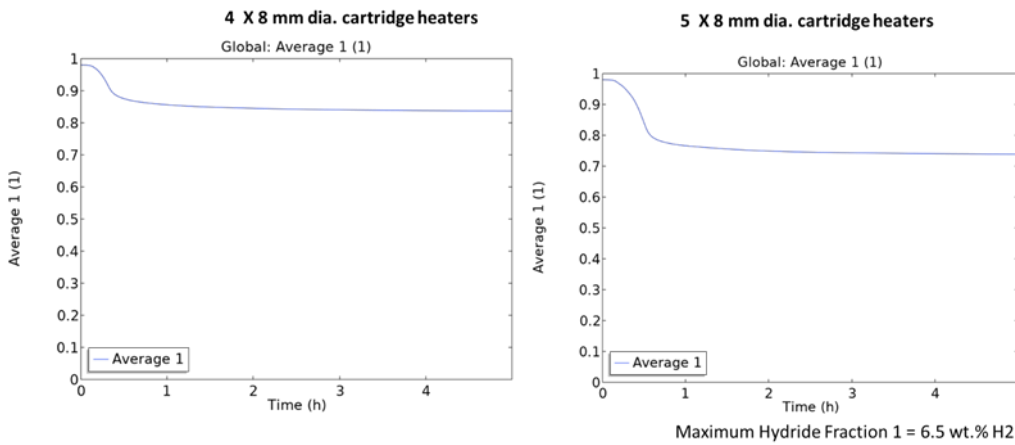Maximum Hydride Fraction 1 = 6.5 wt.% H2

Figure 6 Heat transfer by number of heaters.

Figure 7 shows the heat transfer from the heaters to the media across the particles. As the distance increases in to the media, so the number of potential barriers to conduction also increases, resulting in a rapidly declining temperature.
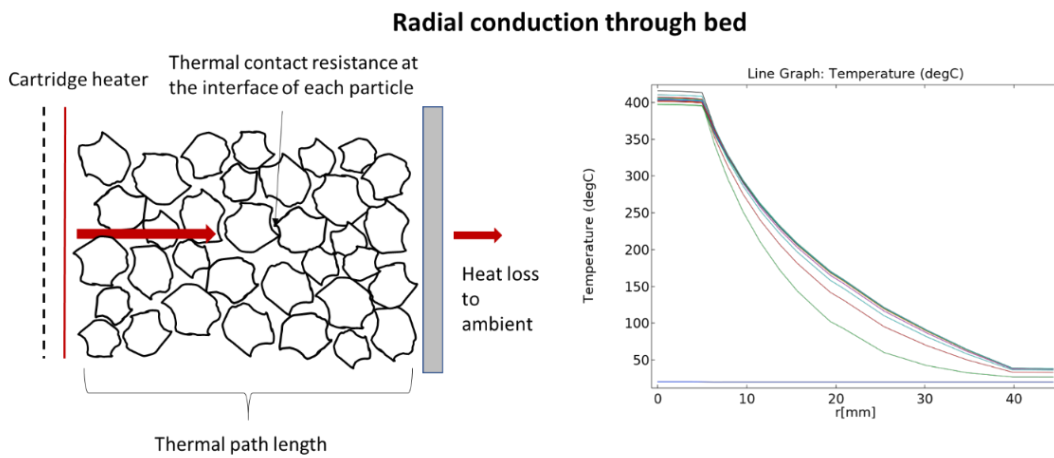
Radial conduction through bed

Cartridge heater

Thermal contact resistance at the interface of each particle

Heat loss to ambient

Thermal path length

Line Graph: Temperature (degC)

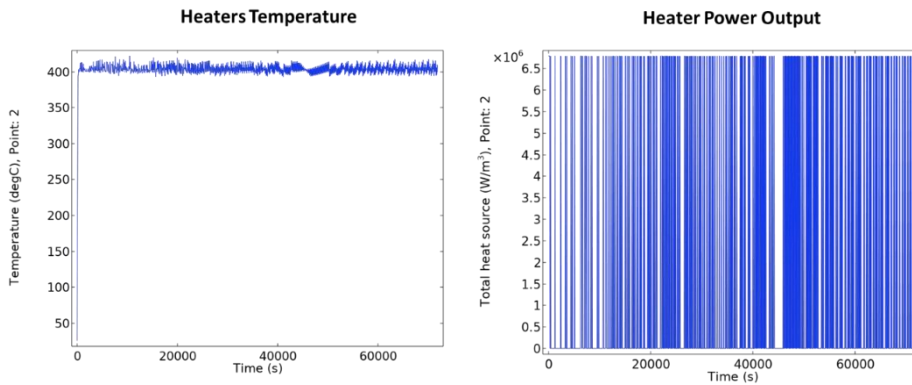**Figure 7** Heat transfer through particles.

Figure 8 Heater temperature and switching times.

In this simulation (Figure 8) when the heater reaches 400°C the heater power is reduced to zero. When the temperature drops below 400°C it is ramped up again. The 'left hand side' shows the overall temperature fluctuation, the 'right hand side' shows the switching times.

4.2.1 Extended surfaces

A method to improve the heat dissipation and promote conduction through the bed is to incorporate extended surfaces. In this case by using longitudinal fins on each heater shown in Figure 9. The fins provide additional conduits through the media.
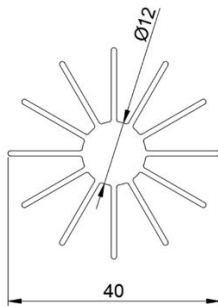

Figure 9. Fin structure with longitudinal fins

The comparison of dehydrogenation improvement from a single fin version shown in Figure 10 and a three fin version shown in Figure 11, shows the central conversion zone has increased dramatically. Figure 12 shows the hydride conversion graphs between one fin and three fins.

Figure 13 shows the comparison between 3 heaters with fins and 5 heaters without fins. 5 heaters increases the rate of heating but the conversion is broadly similar.
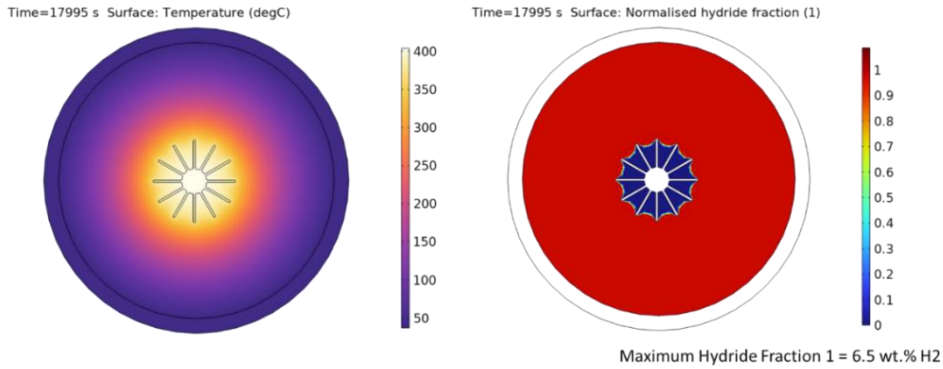
One heater with fins

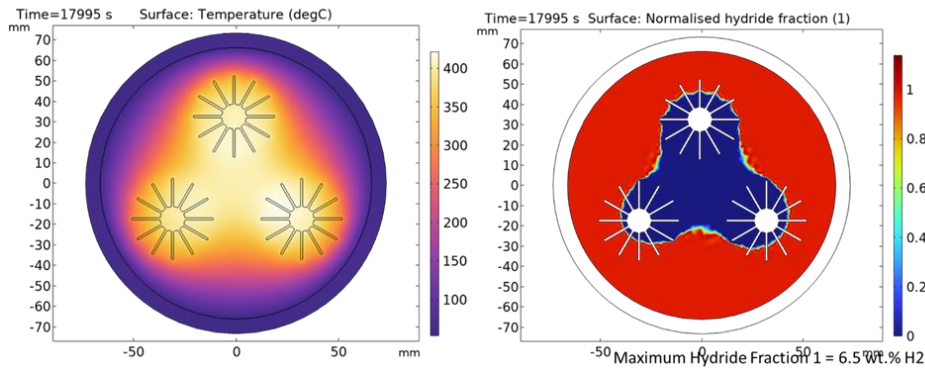Figure 10 Single heater fin thermal profile.

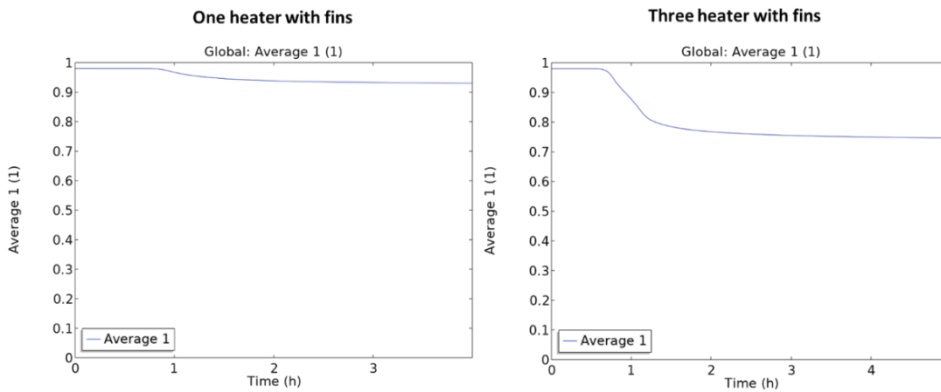Figure 11 Three heater fin thermal profile.

Figure 12.Hydride conversion difference 1 and 3 fins.
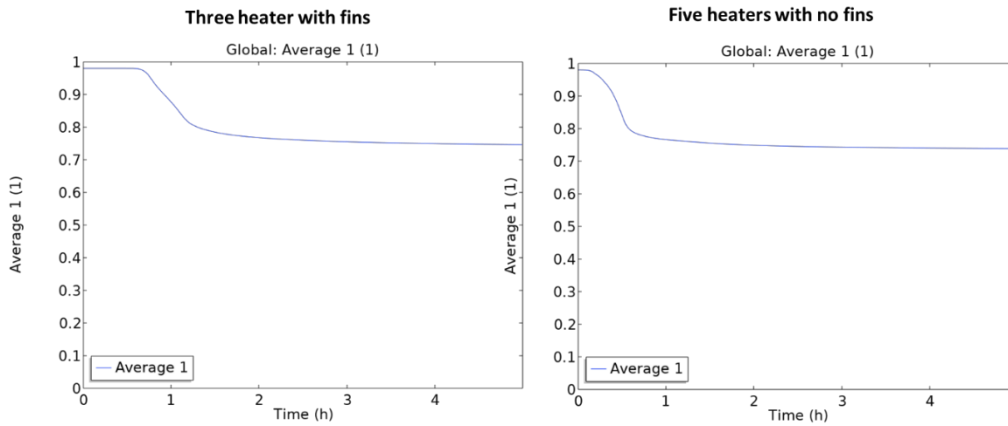
Figure 13 Comparison between 3 fin and 5 heaters without fins.

4.2.2 Heater Location

A second effect of the heater location is the concentration of heat being conducted. A centrally located heater will transfer heat radially through the bed. As the heat is conducted through the bed and away from the heater it will decrease in concentration with radial distance. A more effective approach is to locate the heaters to direct heat from the outer circumference of the store inwardly. This means that as heat transfers towards the centre of the store the heat concentration intensifies elevating the temperature in the material that is surrounded by the ring of heaters shown in Figure 14.

Maximum Hydride Fraction 1 = 6.5 wt.% H2
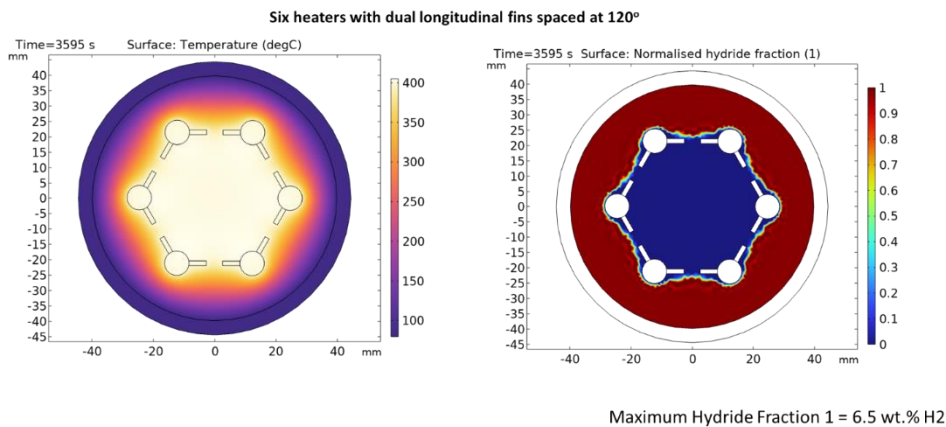
Figure 14 Six heaters with 2 fins.

Figure 15 shows the comparison of 6 heaters and fins to three heaters and fins, where the hydride conversion is improved with 6 heaters.
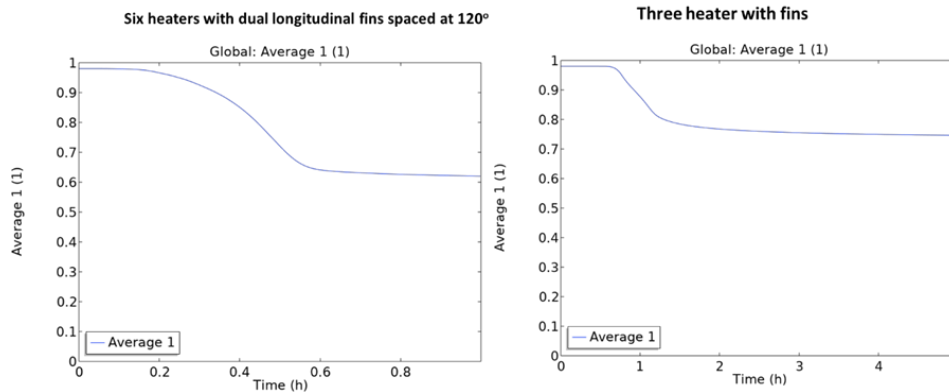
Figure 15 Comparison 6 heaters and 3 heater and fins.

4.3   Experimental Setup

4.3.1   Dehydrogenation Circuit

Figure 16 shows the dehydrogenation circuit comprising the gas delivery and heating system. The gas system does not show the connecting tubes between each component, hence the gaps. A control tap allows gas to be collected, stored or evolved depending on the system conditions. A pressure gauge and pressure relief valve are also provided for safety. The thermocouple feedthrough allows four wires (in this case) to pass through the gas tight fitting and into the various readers. The heater assembly comprises six heaters and heater spreaders to dissipate energy over a wide area. The heaters are located inside the vessel, whereas the gas fittings are external to the vessel.

Figure 16 Dehydrogenation Circuit.

4.3.2   Heat Spreader

Figure 17 shows an expanded view of the heater spreader (end section view). The curved wings (RHS) face to the outside of the cylinder wall, whereas the left hand side helps create a heat front, which travels to the centre of the cylinder. Both parts and the cartridge heater fit through the 18mm diameter vessel orifice.

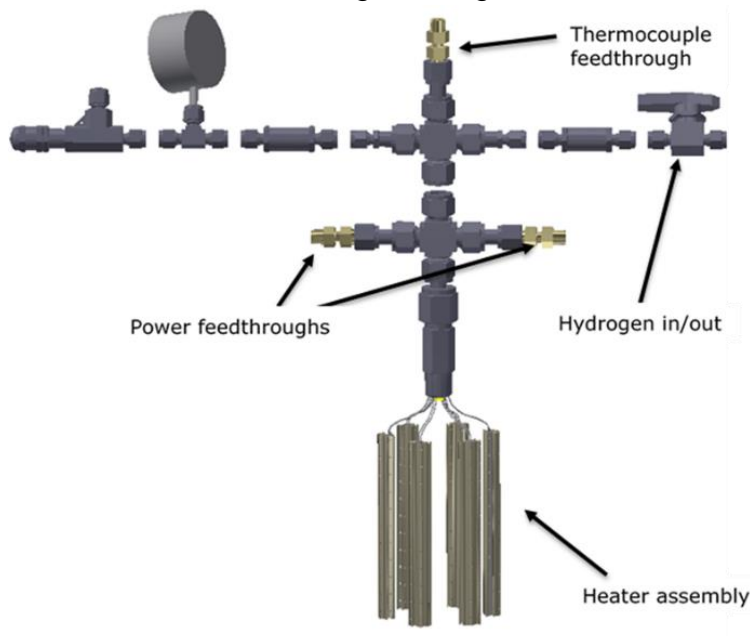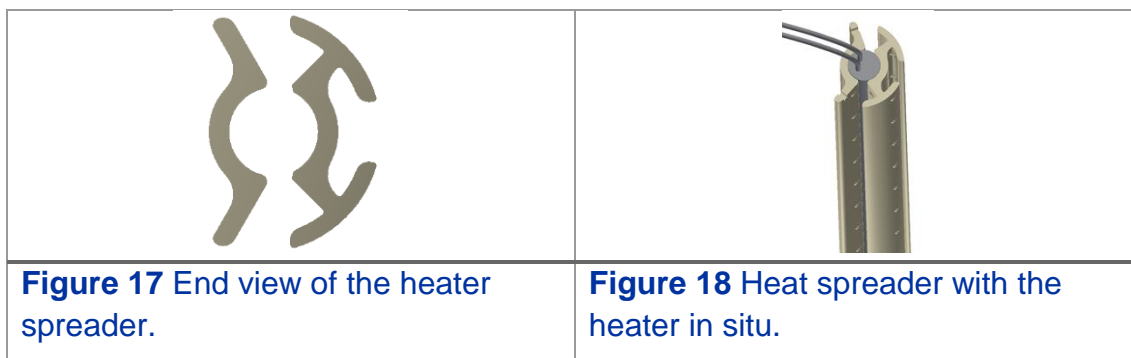|	
Figure 17 End view of the heater spreader.	**Figure 18** Heat spreader with the heater in situ.

The heater shown in Figure 18 is located between the heat spreaders, with an arrayed version of spreaders shown in Figure 19 as assembled inside the cylinder. The nickel alloy has been formed by powder bed additive manufacturing. The holes along the length allow nickel wire to be wrapped through and round the heat spreader, clamping it to the heater. When the powder forms into the matrix the associated volume expansion will further force the components together. The wire loops formed will remain in situ introducing further catalytic nickel and conduction pathways in to the powder.

The positioning of the heater assembly inside the cylinder is important, they are required to be evenly spaced and not touching the cylinder wall. Two thermocouples were located in the initial powder matrix, in order to time the heat transfer centrally to the core. The power input must be controlled to prevent over heating of the matrix. The powder forms a partial matrix by slight melting or diffusion of the metal during hydrogenation. Controlled formation of the matrix at the primary stages is important for system protection and correct conditioning. The individual heaters had a capacity of 350W each, depending on testing results. The payload (Mg powder) was 623.6g excluding heater assembly weight.
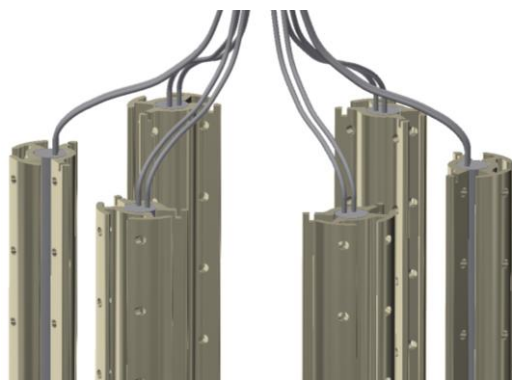
Figure 19 Array of heaters and heat spreaders.

4.3.3   Gas vessel assembly

The gas vessel comprises of a 1L stainless steel sample cylinder supplied by Swagelok, 304L-HDF8-1000. The overall length of the vessel was 277 mm, with an external diameter of 88.9 mm and a minimum wall thickness of 4.6mm. A stainless steel vessel, held vertically, the lower end is partially filled with clean dry silica wool and covers the curved bell radius. The heaters are individually inserted, and two wire crimps are made for the power positive and negative terminal connections. High temperature flying leads were also crimped in place, to make the connection at the opposite ends to the gas tight feedthroughs. The crimps were also wrapped and sleeved in high temperature fibreglass insulation.

4.4   Gas Manifold panel and store installation

The hydrogen store was installed as shown in Figure 20 having a grey thermal jacket. To facilitate the controlled transfer of hydrogen the dehydrogenation circuit is connected to a gas manifold panel, Figure 21. The panel is equipped with two mass flow controllers giving a range of hydrogen flow rates from 0.08 to 65nL/min. The gas manifold is connected to regulated helium, nitrogen and compressed air bench gas supplies, a vacuum pump and a vent line. The inert gases are used to purge and leak test the manifolds and connected store. The vacuum pump enables the manifold and connected store to be pumped down and the vent line enables the safe release of hydrogen externally from the laboratory. The delivery pressure of the hydrogen gas to the store is controlled by hydrogen pressure regulators located on the gas manifold panel.
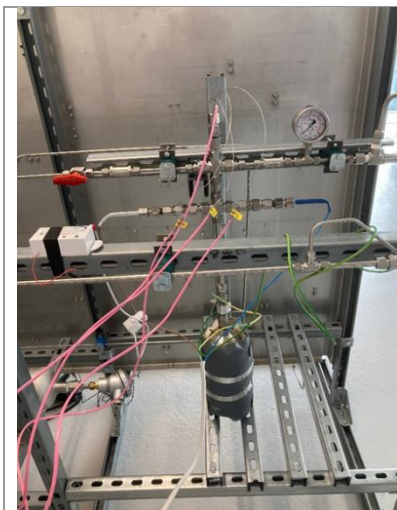
| **Figure 20** High-Store gas containment vessel. | **Figure 21** High-Store gas control panel. |
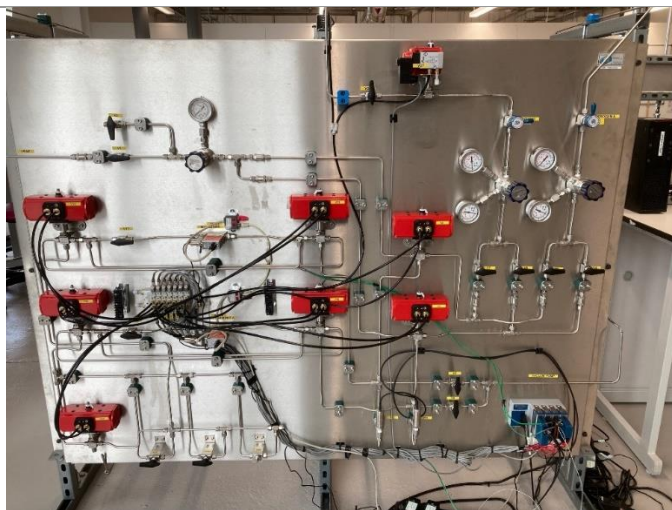
4.4.1 Data Acquisition

A National Instruments data acquisition device (cDAQ) enables pressure, temperature and gas flow rates to be recorded. Thermocouples from the gas assembly are connected to the cDAQ. Data from the cDAQ is logged by a PC running a LabVIEW VI (program). The LabVIEW also controls actuated valves located on the gas manifold panel.

4.4.2 Store Filling with Powder

Prior to filling the vessel with media, the six heaters and connecting wiring were installed inside the vessel through the top inlet. The top assembly was not attached to the sample cylinder at this stage. The store was then placed in a fume cupboard and held in position by a clamp stand. A funnel with a short tube was positioned in the inlet of the store. The short tube was used to ensure that Mg powder was delivered directly into the store without depositing powder on the inlet threads. Powder was measured in a 50 ml beaker then poured into the funnel and repeated several times. The total amount of Mg powder in the store was 623.6g.

4.5 Experimental Results

4.5.1 Activation Process

To activate the Mg powder the sample cylinder was placed under helium gas at a pressure of 19.5bar and heated to 380°C. Once the temperature of the store had reached 380°C the Helium gas was removed. The supply of H_2 gas was set to 18bar on the regulator of the gas manifold panel. H_2 gas was supplied to the store via the mass flow meter on the gas manifold panel. The flow rate was set at 6.5nL/min. Initially as the hydrogen transferred in to the store the flow rate

peaked at 6.5nL/min. After the free space of the store was filled with hydrogen the flow rate dropped to an average of ca. 0.05nL/min. The activation process was stopped prematurely when a software timeout closed the actuated hydrogen supply valve after 23 hours. The capacity reached by Activation 1 was ca. 0.77wt.% H_2, Table 1 and Figure 22. The relatively low final capacity of the store following activation indicates that a large proportion of the Mg powder remained un-activated.

Following the first activation the store was dehydrogenated at a temperature of 380°C and at a pressure of 1 bar. The rate of dehydrogenation was much quicker than the rate of hydrogenation observed during the activation process. The average flow rate of hydrogen gas ca. 0.2nL/min and the final concentration of the store following the first dehydrogenation was ca. 0.2wt.% H_2, i.e., the store was not fully depleted when the first dehydrogenation process was stopped, Table 1 and Figure 23. A second activation was conducted at a temperature of 380°C and an initial hydrogen gas supply pressure of 18bar. After 5 minutes the hydrogen gas supply pressure was increased from 18 to 31 bar to accelerate the rate of hydrogenation and improve the final store capacity. After around two and a half hours the actuated hydrogen valve was activated by a software timeout which caused a period of constant concentration for around 30 minutes. The final store capacity following the second activation process was 2.12wt.% H_2, which was achieved after 21 hours, shown in Figure 22.
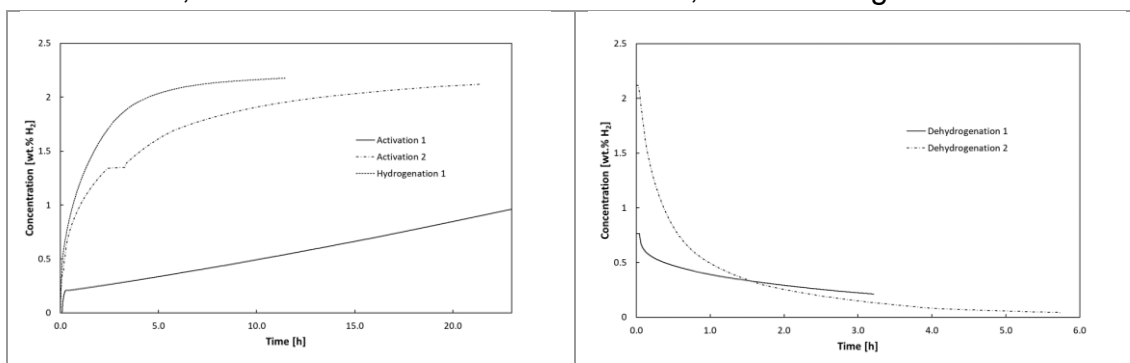
Figure 22 Activation and Hydrogenation Cycles.	**Figure 23** Dehydrogenation Cycles.
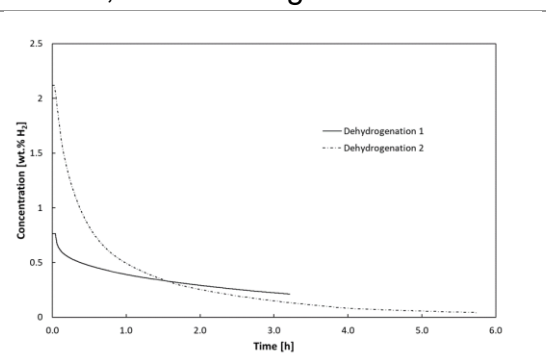
4.5.2   Dehydrogenation

A second dehydrogenation of the store was conducted at a temperature of 380°C and a pressure of 1bar. During dehydrogenation the bed concentration changed from 2.12 to 0.04wt.% H_2, in under 6 hours, shown in Figure 23. The average flow rate was 0.45nL/min H_2, which is an increase of over 100% compared to the previous dehydrogenation which was conducted under the same conditions.

4.5.3 Hydrogenation response time

Following the two activation processes and the two dehydrogenation cycles shown in Table 1, the store was hydrogenated at a temperature of 380°C and at a pressure of ca. 31bar. The hydrogenation process was considerably faster than the activation process with a final concentration of 2.1wt.% H_2 being achieved in ca. 11 hours. The average flow rate of H_2 was ca. 0.25nL/min H_2, which, in comparison to the second activation process, is an increase of 100%. A typical reaction curve for hydrogenation of Mg powder has a very steep initial reaction rate, which flattens out over the final part of the reaction. A similar response is observed in the hydrogenation data of Figure 22, e.g., the average flow rate of hydrogen over the period in which the concentration increases from 0 to 1.7wt.% H_2, was ca. 1.24nL/min H_2. Over the final part of the reaction, in which the concentration increases from 1.75 to 2wt.% H_2, the average flow drops to ca. 0.05nL/min H_2. Although the flow rate towards the end of the reaction is relatively slow, the faster reaction rate at the beginning of the reaction means that three quarters of the stores capacity has been hydrogenated in the first two and a half hours of the hydrogenation process.

Cycle	Initial pressure	Final pressure	Heater Temperature	Initial Concentration	Final Concentration	Average Flow Rate	H2 Transfer	Duration
[-]	[bar]	[bar]	[degC]	[wt.% H_2]	[wt.% H_2]	[nL/min]	[kg]	[h]
Activation 1	19.5	10.5	380	0.000	0.766	0.049	0.00535	23
Dehydrogenation 1	10.5	1.0	381	0.766	0.211	0.207	0.00345	3
Activation 2	18.0	32.0	380	0.211	2.119	0.118	0.0120	21
Dehydrogenation 2	15.0	1.0	380	2.119	0.042	0.450	0.0129	6
Hydrogenation	31.3	31.8	380	0.042	2.170	0.245	0.0136	11.45

Table 1: Experimental Cycles

4.6 Evaluation of Experimental data

4.6.1 Store Capacity

The maximum concentration achieved experimentally following hydrogenation was 2.17wt.% H_2. This equates to 0.33 hydride fraction where 1 equals the maximum practical concentration of 6.5wt.% H_2. The maximum concentration change predicted by the modelling was 0.4 hydride fraction, Figure 14, due to the thermal conductivity of the Mg powder inhibiting heat transfer. Although the experimental concentration of 0.33 is comparable to that predicted by the model. Further investigation is required to determine whether the bed requires additional cycling to allow the working capacity to be fully established. Where 0.4 conversion represents the theoretical maximum conversion, then 0.33 represents 82.5% of the maximum achieved with limited cycling runs. The modelling work for dehydrogenation shows a maximum change in concentration of 0.4 taking place in around an hour, Figure 14.

4.6.2 Hydrogen transfer rate

The modelling of the Mg store predicted that dehydrogenation would be completed within an hour with a fraction capacity of 0.35 of the theoretical maximum, Figure 14. Experimentally the fastest rate of dehydrogenation that was achieved resulted in 75% of the store capacity being dehydrogenated with in 1 hour, as shown in Figure 23.

4.6.3   Challenges to experimental testing

The significant modelling and build requirements including a bespoke control panel and gas control system meant that considerably more resource (approx. factor 2) than was budgeted for was used to deliver the project in the limited time. The build of the prototype store was significantly delayed due to a change in design of the cylinder. The change in cylinder was required as the original cylinder design could not be delivered owing to production difficulties. As a number of components had long lead times this compounded the difficulties already caused and further reduced available experimental time.

Despite these challenges the team worked well together delivering and testing a prototype in a limited time. The experiment was therefore to show that the expected fraction capacity of 0.35 for this small diameter vessel would be achieved. Since practical Mg hydride has a tested working capacity of 6.5wt. % hydrogen delivered by the magnesium hydride in the vessel (opposed to theoretical 7.6wt %) then this equates to a target capacity of ca. 2.3wt%. As can be seen the two cycles achieved over 2.1wt%. With further cycling typically it would normally be expected that the hydride fraction would increase further (typically taking at least 10 cycles) so this was an encouraging result.

5   Phase 2 demonstration project

5.1   Phase 2 Objectives

Building upon the experience gained in phase 1, a demonstrator system will be designed and built in Phase 2 to meet the following experimental objectives:
- Demonstrate the ability of the store to operate (hydrogenate and dehydrogenate) under different pressures in the range of 8 to 30bar.
- Achieve a storage centration of between 1339 and 2054kWh/m^3 (2/3 of 6.5 and 6.5wt.% H_2 respectively).
- Achieve a total heat input of 14kWh per kg H_2 (associated emissions 336g CO_2 per kg H2).
- Identify, in conjunction with cylinder manufacturer, the dimension of the store (diameter/length) that would provide the most efficient storage solution whilst remaining commercially viable.
- Determine experimentally the level of impurities, which can be tolerated by the Mg particles and whether the cycling of the store provides a mechanism by which impurities can be removed.

- Provide an in-depth analyses of the LCOS of counterfactual storage and compare with both the static commercial storage solution together and the multiple element gas container concept.
- Provide analysis of the potential of High-Store to provide load smoothing capability to help balance renewable fluctuations.
- Conduct a LCA of the preferred storage option as determined by objective 6. The LCA would identify the lifecycle emissions associated with the provision of hydrogen storage.

5.1.1 Building Upon Phase 1

- The following practical measures identified in phase 1, will be accommodated in the design of the phase 2 demonstrator: The vessel is required to have a wide port about 85% of the vessel outside diameter and wide bodied cylinder to maximise capacity.
- Insulation is required for the internal vessel wall, vermiculite has been shown to have a compatible chemistry but requires investigation.
- Source heaters with higher level of thermal resistance, durability and connectivity.
- Locking heaters into a preformed matrix, would develop a resilient construction approach.
- Heater zones offer a way to control total power by time; using vertical segregation.
- Considering scaling the application to road transportation.

5.2 Phase 2 Markets Evaluation

The concept of the Multiple element gas container (MEGC) will be investigated and a potential business case developed.

Building upon the storage solution established in Phase 1, a more extensive review of the potential commercial uses will be conducted in Phase 2. This will include the following:

- Further development of the multiple element gas container concept (MEGC) which is potentially a cost effective way to increase the scale of vessels used.
- Investigate the use of MEGCs for both static and transport options. Potentially MEGCs offer a good choice to scale static and transport solutions simultaneously, whilst offering a practical solution to the challenges of providing electrical control systems.
- Explore the business case for end users who would have existing electrical supply.
- Explore a wider range of potential markets including:
  - Heavy shipping ballast tank replacement.

- o Ferry operations (drive-on drive-off replacement).
- o Road and rail container transportation.
- o Sensitive environments where high pressure cylinders may be excluded.
- o Load smoothing renewables without compression losses.
- o Long term storage/usage.

5.3   Description of Phase 2 MEGC concept

Phase 2 will build a version of an MEGC to hold several containment vessels together. Each vessel will consist of a wide tube type cylinder with a wide orifice through which the following parts will be placed:

- Insulation.
- Heater matrix assembly with heaters and wiring all held in situ. The heaters will contain more than one layer of heaters
- Magnesium powder fill.
- Optional extra (possible second iteration) may include hot gas pre-heat coil from other waste exhaust etc.
- Seals including electrical and sensor feedthroughs.

A range of vessels will be incorporated into a steel framework. The framework and vessels (MEGC) will provide the platform from which experiments will be conducted. The MEGC will contain part of the electrical control system that provides the electrical inputs.

The electrical control system will quickly switch between heating levels and cylinders to maintain a suitable gas supply. It will have to predict likely thermal fluctuations based on historic learning. The MEGC will be of a size (initially) where the individual cylinders can be hand carried even if the whole unit cannot.

The storage volume on a volume by volume basis of the media should be comparable to the storage volume of liquid hydrogen. But as the High-Store hydrogen is made from renewables there is zero CO_2 produced, there is no gas compression, again saving the CO_2 1.3kg per 1kg of hydrogen traditionally compressed to 700barg.

The CO_2 produced by the heater's electrical consumption has not been accounted for in the 1.3kg CO_2 per kg compressed H_2. A figure of 2.26 kgCO_2 per kg H_2 has been calculated, due to the heat demand for dehydrogenation in High-Store. This means if waste heat is not available for the High-Store operation then in comparison to compressed storage an additional 0.96 (2.26 – 1.3) kg CO_2 per kg H_2 would be generated by High-Store. However in a road transportation operating scenario where a lorry is traveling 400km (200km each

way), the saving is 4.43 $kgCO_2$/kg H_2. This scenario considers the movement of a traditional cylinder, again it does not include the CO_2 generated by the electric heaters. When the electrical heating is considered (where there is no waste heat available) the CO_2 saving reduces to 2.17 $kgCO_2$ per kg H_2.

The failure of a high pressure gas cylinder at 700barg could be viewed as an explosive occurrence with possible fragmentation of nearby articles. By comparison the failure of a High-Store MEGC where the units are at a low pressure, containing a low free volume of hydrogen, would just result in a limited escape of gas.

6   Design of Phase 2 demonstration store

The phase 2 vessel design would look similar to that shown in Figure 24 (note IP sensitive fins and configuration not shown) however there may be more than one layer of heaters. The heaters may be embedded in a metal heat fin arrangement, which holds all the internals together. The heat fin structure would then be filled with the powder.
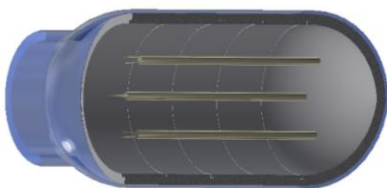

Figure 24 Section view of vessel, showing vessel wall, insulation and half of the heaters.

The phase2 project would develop a framework structure where 6 vessels are held vertically. Each vessel being 15 to 20kg when filled (nominal). By linking several vessels in close proximity within a heavy duty frame and one control system which operates all the vessels as required. This would represent a High-Store version of a commercial MEGC but at smaller scale, with a total weight of less than 170kg.

The work package structure consists of:
WP1 Project Management – TWI (£416k). WP2 Simulation – UoN (£443k).
WP3 Power – J4IC (£360k). WP4 Vessels – CSC (£388k).
WP5 Internal structures – TWI (£416k). WP6 Commercial – CSC (£222k).
WP7 Assembly testing and results – UoN (all) (£526k).

7   Benefits and barriers

7.1   Benefits

When comparing High-Store to traditional cylinders for a transport movement i.e. for a delivery to a site, the carbon savings have been estimated at 4.43$kgCO_2$/kgH_2.

The reaction between Mg powder and hydrogen means that the gas is stored as a solid. This reaction is highly reversible and relies on the provision of heat to release the hydrogen. If waste heat is available the efficiency of the process can be improved. Phase2 has incorporated an experimental tube coil to recirculate waste heat in order to prototype the theory.

Once hydrogen has reacted with the magnesium the resulting metal hydride is stable at room temperature. This means that in the event of a mechanical failure or rupture of the enclosure, the magnesium hydride will not spontaneously combust nor will large amounts of hydrogen gas be released. The media can be cycled thousands of times without capacity loss. UoN have demonstrated this function on small Mg samples.

High-Store can operate on supply pressures as low as 10 bar and generate gas delivery pressures of 5 to10 bar. This provides a mechanism by which the need for gas compression can be removed from a system improving overall efficiency, increasing reliability and reducing operating costs.

The use of Mg powder to store hydrogen does not introduce further impurities into the hydrogen. Refuelling comes directly from electrolysers at the pressure produced. The hydrogen is pure, dried and ready for storage. An approximate rule of thumb for magnesium hydride storage is that 15 tonnes of magnesium will store approximately 1 tonne of hydrogen; excluding vessels and equipment. Therefore lorries should be able to carry over 1 tonne at low pressure. Whereas static storage is only limited by free land and the ability to recharge or condition the store.

7.2   Challenges

7.2.1   Research Challenges

Building on the initial testing conducted in Phase 1 there remains a number of experimental tests that need to be conducted in Phase 2. These include demonstrating the store operating under hydrogenation and dehydrogenation conditions but under pressures in the range of 8 to 30bar. Achieving a storage capacity between 1339 to 2054kWh/m^3 (2/3 of 6.5 and 6.5wt.% H$_2$ respectively).

7.2.2   Design Challenges

A vessel redesign is needed this will require approvals, in order for it to be used, even for research testing. A heat spreader metal matrix is required which holds the heaters. This could be a single part but it's more likely to be made up from extrusion pieces. A components-off-the-shelf approach will be adopted for the

controller system, it being located with the MEGC to form a modular demonstration package.

A new vessel requires new vessel gas connections, it will be required to use gas tight feedthroughs. While it was proved that commercial fittings could form helium leak tight unions, there could be a risk that new fittings would be untested.

7.3 CapEx, OpEx, Costs and Counterfactual scaling.

7.3.1 H2 Capacity of commercial static solution

The CapEx and OpEx costs have been determined for static installations comprising of 100 modules each containing 6 x 300L cylinders i.e., a total of 600 cylinders. As each cylinder will include a heater/fin assembly and an expansion allowance the mass of Mg powder per cylinder is 225kg. Based upon a working capacity of 6.5wt.%H_2 (representative of a practical working capacity) the mass of H_2 stored per cylinder will be 15.6kg. If the installation provides a weekly operation utilising its full capacity then the total annual storage of H_2 is approximately 19228MWh. 15.6 x 600x 52x 39.4kW/kg.

7.3.2 Heating Requirement of commercial static solution

Using the theoretical enthalpy of formation for MgH_2 the heating requirement is 10.2kWh/kg H_2. If the electrical heating is assumed to be 80% efficient then the electrical energy input for dehydrogenation is 5973GW. The heating requirement for hydrogenation is assumed to be 1/3 of the dehydrogenation due to the reaction being exothermic then the total annual heating requirement is 7944GWh.

7.3.3 Annual Operating Costs

Total annual OpEx is £1.67m. for the static version.

7.4 Levelized cost of hydrogen

The levelized costs are provided in Table 2.

Metric	Units	HighStore Exp (2.17 wt.%H_2	HighStore theoretical working capacity 6.5wt%H_2	Compressed H_2 at 700 bar	Liquefied H_2
Volumetric capacity	[kg/L H_2]	0.0135	0.041***	0.04	0.071
Energy requirement	[kWh/kg H_2]	-	10.2	6	11 to 28

| Energy requirement | [kWh/kWh H_2] | - | 0.26 | 0.15 | 0.28 to 0.71 |
| LCOS | [£/MWh H_2] | - | 435 | ?** | 175* |

Table 2 Levelized costs

*https://assets.publishing.service.gov.uk/government/uploads/system/uploads/attachment_data/file/1011506/Hydrogen_Production_Costs_2021.pdf Page 29 - approximation of upper figures for electrolysis.
** Figures not available or highly variable.
*** 0.05 ideal maximum case for magnesium

7.4.1   Comparison with Counterfactual

A comparison with state of the art, above ground compressed H_2 storage, is not easily accomplished. The cost of storage is rapidly changing depending on supplier and context. Figures given in the public domain range from 50 to 600£/MWh which highlights the difficulty in obtaining accurate comparative data. Examples of some of the uncertainties which exist in the comparison include:

- The method of hydrogen generation has an impact on whether it can transfer directly into the store.
- If there is any available electricity or waste heat for heating the store will impact the emissions associated with the hydrogen.
- Determination of the operating costs of mechanical hydrogen compressors (this is difficult to obtain accurately due to the small numbers of pilot scale refuelling stations where OpEx costs are not always transparent).

A reliable comparison would be a deliverable of the Phase 2 project analysis. The scaling against a counterfactual which are traditional pressurised gas cylinders in this case, is eminently possible. The High-Store vessels offer the capacity to carry twice as much hydrogen, without the CO_2 penalty. The implementation can be scaled, the costs for the first in kind have been documented at £214k for a large scale static 1200kg hydride store comprising 6 vessels, and a lorry flatbed MEGC version at £414k inc flatbed.

A rough rule of thumb that is often used is the vessel including fittings is usually approximately similar weight as the metal hydride within it. Therefore a 1 tonne hydrogen store based on 6.5wt.% hydrogen metal hydride will have a weight of approximately 15 tonnes of powder excluding fittings and vessel, but occupying approximately half the volume of a compressed gas store (counterfactual). It is often quoted that the vessels have to be long and thin for effective heat management. This is exactly the point the research is trying to by-pass by introducing an internal heating network and fin arrangement. This allows for

shorter and fatter vessels with larger diameters. These will be much more cost effective and the weight penalty of the vessel will be greatly reduced.

The challenge for stage 2 is to answer the question - can the store be dehydrogenated in a practical time. The dehydrogenation is an endothermic reaction, therefore, heat has to be supplied through the electrical heater network inside the vessel. It is assumed that a renewable source of electricity is used at this stage. For the phase2 hydrogenation stage the level of cooling required will be investigated. Storage at low pressure is also beneficial to prevent the consequences of leaks, required blast zone distances as well as the cost and weight of vessels.

7.5   % purity of hydrogen

Five nines hydrogen is readily achieved with metal hydride stores. This is because only hydrogen is hydrogenated in the metal hydride and only hydrogen is released. Thus any impurity in the source gas is not trapped by the material or if it is, it is not released. Depending on the source of hydrogen then impurities may poison the store. Hydrogen from PEM electrolysers has been used at the UoN laboratory for initial trials. Other sources of green hydrogen from bio-waste or types of electrolyser will have impurities that will need to be tested to determine what level of poisoning occurs and whether purification is needed. Oxygen impurities can be regenerated. The influence of impurities on capacity on a larger scale store will be investigated in phase2. It is not anticipated that there will be a build-up of impurities in the gas phase due to the cycling of the store and release of gas when dehydrogenating will effectively cause a flow of gas diluting any impurities in the head space accumulated when hydrogenating the store. This will be examined in stage 2.

7.6   The greenhouse gases mitigated

$4.43 kgCO_2/kgH_2$ saving, against a traditional cylinder being transported 200km.

8   Costed development plan

The research necessary to reach the first demonstration units is £9.5m being: Phase 1 (current phase) to provide a small-scale single unit technology demonstrator. Phase 2 £3m and 2 years (next application) to provide a small scale MEGC type solution, with integrated electronic control systems, and using internally insulated vessels. Phase 3 £1.5m and 1.5 years. To increase the capacity of MEGC and incorporate multiple heating zones in each cylinder. Integrate a commercial control system in a fully representative rack.
Phase 4 £5m and 3 years; Delivering 1st in kind full sized system as production representative samples: Product 1 'Static' 12 months. Product 2 'Transportable' single pod 18 months. Product 3 'Transportable' multi pod (MEGC) system 30

months. The first in kind includes the CapEx costs for the static solution (100 units of 6 cylinder modules).

9   Rollout potential

The High-Store technology rollout potential is high, the MEGC product has been chosen as a demonstrator because of the adaptability of the form factor. Therefore, it can meet many needs or customer requirements by scaling the number of vessels, or by changing the size of the vessels. Chesterfield Special Cylinders (CSC) offer these products (traditional cylinder designs) to a vast number of sites and usage cases.

By comparison to the counterfactuals of traditional cylinders, where the industry requirement for increasing storage capacities results in higher pressure storage, resulting in increasingly robust and heavy cylinders which becomes its own rate determining limitation. As the cylinders transition from 350 to 700barg, the storage is accompanied by increased pressure risks, and the fact it will not be possible to add more pressure. With further research it should be possible to reduce the High-Store storage pressure to less than 30barg. Current trials have been run at 30barg to provide an overpressure to the reversible storage reaction, while this is a comparatively low pressure, with additional work it may be possible to reduce the operating pressure to nominally 10barg. Whether the High-Store pressure is 30 or 10barg, it should be possible to operate larger storage vessels than the counterfactual; by extrapolation future single vessels could possibly hold 1 tonne of hydrogen (static version).

Even though High-Store has the potential to store more hydrogen in much larger vessels than is currently achievable, the TRL is untested, and because the energy requirement to recover the hydrogen is likely to be high, the use of some form of waste heat would be beneficial. The waste heat being used to cause either a preheating or near full capacity heating of the contents, and by using electrical heating to top-up or provide fine adjustment of the total heating capacity. Therefore the energy requirement could be dramatically reduced. Phase2 will start the investigation and physical trial of using waste heat in conjunction with electrical heating.

While static vessels can be limited to road weight limits, they can essentially be larger than road going hydrogen transporters, which are covered by different regulations, these weight limits and vessel specifications will be investigated as part of phase2. With respect to road haulage hydrogen transportation systems CSC believe they could convert 1 customer immediately, with a second to follow reasonably quickly. By 2030 CSC could convert 6 to 7 customers to High-Store, capturing 70% of the UK fleet market. As the market continues to expand in the remaining years to 2037, volumes are likely to double during that period.

10   Route to market assessment

The research and development phases to reach a commercial product have been documented. A portfolio offering of High-Store products compliments CSCs existing product lines, by offering increased capacity and safety. These are particularly pertinent to locations where it would be unwise to store high pressure vessels currently, or where regular low pressure recharging avoids gas compression losses. The technology fits well to a number of markets and scales easily. It naturally fits to the green agenda and net zero emissions, especially when charged directly from renewables generation.

New jobs will be created for this exciting development involving the following: Buildings, Manufacturing: Supplier components, Unit supply, Standard products team and Bespoke products team. Installation companies, Software development, Software customisation, Transport systems, Flatbed manufacturing, Servicing, R+D, Sales, Management, Contract and support services. The carbon saving potential has been determined as $4.43 kgCO_2/kgH_2$ saving, against a traditional cylinder being transported 200km.

Current hydrogen production is approximately 0.7Mt per year (UK).
https://cms.law/en/int/expert-guides/cms-expert-guide-to-hydrogen/united-kingdom#:~:text=Industry%20sector%20use,gas%20(i.e.%20grey%20hydrogen).
Therefore High-Store could save roughly 3Mt of CO_2 per year.

11   Dissemination

The High-Store public press release was disseminated through the BEIS website and its own press release.

12   Conclusions

The final design of the dehydrogenation circuit has been presented and tested. During the project the design has undergone many revisions to meet the ever-changing conditions both of the project simulations and amendment of the cylinder conditions.

The results have validated the approach, even though it wasn't possible to fully condition the hydride, due to time limitations only 20% of the necessary conditioning runs were possible. However the dehydrogenation returned 82.5% of the simulated result. With further conditioning the results would continue to improve.

The findings from the feasibility study show that hydrogen can be stored as the hydride form and that this is a low energy process. On recovering the hydrogen, it is clean and dry, ostensibly regarded as pure hydrogen 99.99+%. While the storage of the hydrogen is a low energy process, the recovery (dehydrogenation) to reform the hydrogen in to gas takes more energy. It has been calculated this would be approximately a third of the energy contained within the hydrogen. It has also been hypothesised that phase2 could demonstrate a secondary fluid heating circuit (gas or liquid) which could use waste heat to either partially or fully promote the dehydrogenation of the media, thereby saving some or all of the required electrical heating energy. If it were possible to demonstrate this technology advancement then by comparison to counterfactual the energy of compression is saved, compressor servicing is eliminated, and High-Store operates at safe pressures. Saving 1.3kg of CO_2/kg of hydrogen if the counterfactual is compressed to 700bar.

If hydrogen is liquefied the amount of hydrogen stored in a volume is given by 0.071kg in 1 litre or 0.071kg/l H_2. By comparison the likely maximum High-Store could achieved is 0.041kg/l H_2. For the first experiment 0.014kg/l H_2 was achieved. The absolute maximum which could be achieved is 0.05kg/l H_2. The phase2 would initially build towards $^2/_3$rds of 0.041kg/l H_2.

While the volume of liquefied hydrogen is 0.071kg/l the figure does not account for the amount of insulation and other factors which are required to keep the liquid cold and minimise boil-off. As the insulation which is potentially lightweight, adds considerable bulk to a system, this has not been accounted for in the calculations.

The next phase should also consider producing wide opening cylinders through which insulation and a suitable heat transfer matrix could be quickly assembled. Software will be used to simulate the boundary conditions for best use of materials and thermal loss reduction. Electrical controls should consider algorithm development for monitoring and control of heaters to pre-empt condition changes, also providing the ability to rapidly switch between heater zones to cause homogenous heating but with the lowest overall instantaneous current supply.

To that end, various test units will be produced leading to a final MEGC demonstration unit.