

## 5 L급 액체수소 저장용기의 성능특성 연구

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### Performance of a 5 L Liquid Hydrogen Storage Vessel

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**Abstract** >> In the face of the world's growing energy storage needs, liquid hydrogen offers a high energy density solution for the storage and transport of energy throughout society. A 5 L liquid hydrogen storage tank has been designed, fabricated and tested to investigate boil-off rate of liquid hydrogen. As the insulation plays a key role on the cryogenic vessels, various insulation methods have been employed. To reduce heat conduction loss, the epoxy resin-based insulation supports G-10 were used. To minimize radiation heat loss, vapor cooled radiation shield, multi-layer insulation, and high vacuum were adopted. Mass flow meter was used to measure boil-off rate of the 5 L cryogenic vessel. A series of performance tests were done for liquid nitrogen and liquid hydrogen to compare with design parameters, resulting in the boil-off rate of 1.7%/day for liquid nitrogen and 16.8%/day for liquid hydrogen at maximum.

**Key words** : Liquid Hydrogen(액체수소), Cryogenic Storage Vessel(극저온 저장용기), Insulation(단열), Boil-off Rate(증발율)

### 1. INTRODUCTION

Today's world has large energy needs. These needs are growing especially due to the emerging and industrializing nations such as China or India. The world also

faces a limit on non-renewable energy resources, such as oil. In addition, there is the large more pressing issue of global warming on the horizon caused by the consumption of these non-renewable sources i.e. coal, oil, or natural gas. These issues have forced us to look into alternative sources of energy ranging from solar, bio-energy, hydroelectric, or geothermal. Storing and distributing this produced energy is important to the utilization of these

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emerging technologies<sup>1)</sup>. Hydrogen can offer us one of the many possible answers for energy storage and usage.

Hydrogen is the most abundant element in the universe and is available in vast amounts on earth. Production of hydrogen today is generally done through steam methane reforming of fossil fuels. While this method is non-renewable, future production methods such as high temperature water splitting, solar electrolysis, or biological reactors offer us renewable methods for creating hydrogen<sup>2)</sup>. Once produced, hydrogen can then be converted to mechanical energy by combustion in an engine or utilized in a fuel-cell to produce electrical energy. It is the least polluting fuel available; it is CO<sub>2</sub> free (when produced via renewables), limited to no NO<sub>x</sub> content, and water is the only by product.

To effectively use hydrogen as a fuel, it must be able to be stored. To store hydrogen, there are currently several possible methods: compression of gaseous hydrogen, metal hydride and liquefied hydrogen etc. Each of these has their benefits and drawbacks. Storing as a compressed gas can easily be done today with existing compressors but it is hazardous due to the extremely high pressures needed to create a good energy density. Metal hydride while safer the energy density is lower than the other options. Liquid hydrogen (LH2) storage requires various advanced insulation techniques to maintain it in a cryogenic liquid state and reduce boil-off (vaporization of LH2); LH2 storage offers a low pressure high energy density method for the storage of hydrogen<sup>3-8)</sup>. Advancing the development of the insulation techniques is a major concern for the use of LH2.

Recognizing the need for improving cryogenic liquefaction and storage technology, Korea Institute of Science and technology (KIST) began research and successfully demonstrated in 1997 a small scale liquefier and storage

tank<sup>9)</sup>. This research continues today at KIST; this paper describes system design, fabrication method and experimental results of a 5 L storage tank, with more advanced technologies than before. The hydrogen liquefier and the catalysts for ortho-para hydrogen conversion have been also studied at KIST as well<sup>10-11)</sup>.

## 2. Design, Fabrication, and Experimental Method

### 2.1 Experimental Design

The 5 L storage tank was designed with target boil-off rate of 0.11 L/day under liquid nitrogen (LN2) and 0.57 L/day under liquid hydrogen. To effectively design a cryogenic storage tank, one has to take into account various sources of heat leak. Usage of a vacuum shield or shell minimizes the convective heat transfer to the inner tank. This tank was designed to use both a mechanical vacuum pump and a turbo-molecular pump (TMP) to achieve 10<sup>-4</sup> Torr or lower in the shell. Wrapping multi-layer insulation (MLI) around the inner tank coupled with the vacuum shield significantly reduces radiation heat transfer. This tank was designed to use double sided aluminized Mylar (DAM) MLI with a glass paper insert to effectively insulate against this radiation heat transfer. The use of a vapor cooled shield (VCS) was also incorporated into the design; a VCS removes radiation heat penetrating through the MLI. The last source of heat leak is through conduction. Use of low thermal conductive materials with long thermal conduction paths is necessary for a good design. The design used two, 1 meter G-10 pipes to support the 5 L storage tank. G-10 is a low thermally conductive epoxy resin embedded with fiber glass<sup>12)</sup>. Further detailed explanation and calculations of how various design parameters, such as length of G-10

rods, diameter of the radiation shield or aspect ratio of the inner storage tank, affected the calculated heat load will be presented in an upcoming paper.

## 2.2 Fabrication and Assembly of 5 L Storage

Using design data obtained from theoretical thermal calculations, fabrication of the 5 L vacuum insulated cryostat began. Outer shell was fabricated with 304 Stainless Steel (304 SUS) and the lower shell upper flange was machined with a groove for a Viton O-ring (Fig. 1).

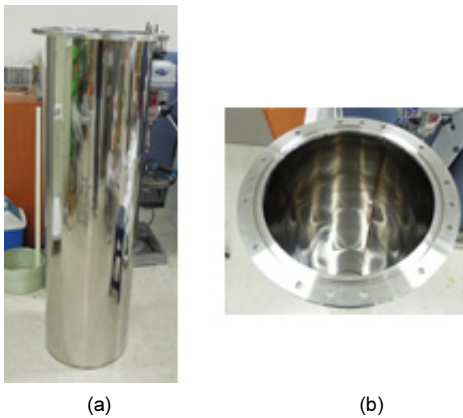


Fig. 1 Outer lower shell; (a) final fabrication and (b) flange.

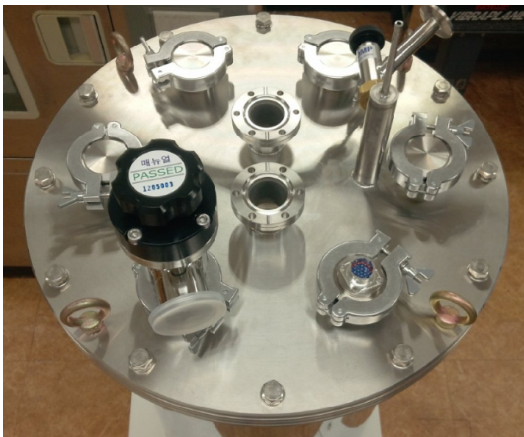


Fig. 2 Top Flange of outer shell

The upper flange equipped with 6, NW-40 vacuum ports for sensors, evacuation and other uses (Fig. 2). Two 2.75" OD CF flanges were welded in the center of the upper flange for a liquid filling bayonet setup and an optional liquid level sensor. One vacuum and MLI jacketed off gassing line was also installed.

The 5 L container was fabricated using 304 SUS. The 5 L tank contained 3 holes in the top: one for filling/draining, the other for liquid level sensor and a 1/4" pipe for boil-off gas. To connect and support the 5 L container two G-10CR pipes were used (Fig. 3). Both G-10CR pipes were threaded on both ends. One end threaded into a machined fitting welded to the top of the 5 L container; the other end threaded into a machined G-10 flange with an O-ring groove. The G-10 pieces and SUS tank were connected and sealed using Lakeshore's cryogenic low off-gassing Stycast epoxy. After the epoxy cured, a Viton O-ring was put into the groove on the G-10 flange and mounted to the inside surface of the upper flange. The structure was mounted in the center with the G-10 pipes/flanges aligned with the holes for the two external CF flanges.

Next piece, the vapor cooled shield (VCS), was fabricated (Fig. 4). The shield is made of 2 mm copper

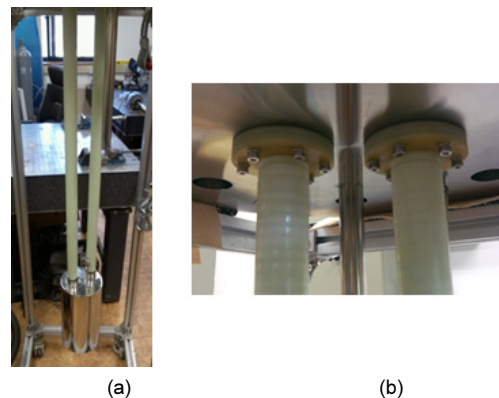
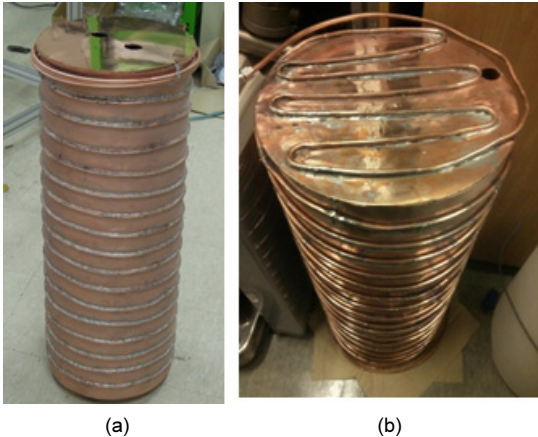
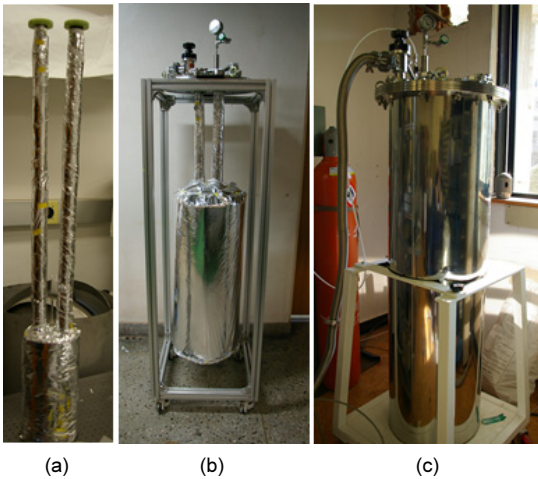


Fig. 3 Internal structure; (a) final fabrication and (b) mounting to the upper flange



**Fig. 4** VCS final fabrication; (a) side-top view and (b) bottom view



**Fig. 5** (a) MLI wrapping internal structure, (b) vacuum shield and (c) final assembly

sheet. After fabricating the base copper shield, 1/4" copper tube was then wrapped around it and soldered to the surface to improve thermal contact. The shield is able to be hung from the lid of the outer shell using 8 kg-test monofilament fishing line.

Before hanging the VCS, double sided aluminized Mylar (DAM) MLI with glass paper spacer insert was used to wrap the 5 L tank and G-10 pipes 40 layers. After wrapping, the VCS was hung and connected to the

boil-off gas port on the 5 L tank. The boil-off gas will flow from the bottom of the shield to the top before it exits through the vacuum jacketed exhaust line. 40 layers of DAM MLI were also applied to the outer surface of the radiation shield. MLI was wrapped to cover all metal surfaces up to the vacuum jacketed exhaust line. The internal structure was then inserted into the outer shell and sealed (Fig. 5).

## 2.3 Experimental Method

### 2.3.1 Setup and Instrumentation

After completing full fabrication of the cryostat, to monitor vacuum level, a Convectron vacuum gauge was installed on one of the NW 40 ports on upper flange of the cryostat. The cryostat was then hooked up to a mechanical vacuum pump. The tank was evacuated, and purged with dry nitrogen. The purging was done at least 3 times to remove any oxygen, humidity and any moisture adsorbed into the MLI. After the purging cycle, the tank was evacuated until a maximum of 5mTorr is reached. The mechanical vacuum pump ran continuously for the duration of the experiments. A turbo-molecular pump was not available for instillation during these experiments. There will be expected high thermal leak due to lower vacuum level.

To measure boil-off rate, the vapor exhaust line was hooked up to an Omega FMA-1601A 20 SLPM mass flow meter (MFM). The MFM was wired into a National Instruments Compact Field Point system and then the flow data was recorded via LabView. Fill volume was measured with an American Magnetics liquid level sensor. This sensor was calibrated in a liquid nitrogen Dewar before the start of the experiments

Standard safety precautions were taken for working

with both cryogenic liquids and explosive gases. For explosive gases, the experiments were conducted in a well-ventilated room under a large walk-in fume hood. Hydrogen sensors were installed both on the ceiling of the room outside the fume hood and in the fume hood. In the case of power outage and gas leak, these sensors would trigger an audio-visual alarm outside of the room indicating the leak. All electrical connections and monitoring equipment took place external to the fume hood to prevent any possible ignition sources. For cryogenic liquids, appropriate safety equipment was used; cryogenic gloves, full face shields, and long sleeved clothing were worn to prevent any direct contact with any cold surface or splashing when handling the liquid nitrogen.

### 2.3.2 Starting and Running the Experiments

Liquid nitrogen was transferred slowly from a standard LN2 storage Dewar into the 5L tank via a vacuum insulated transfer line. Once the internal tank was full, the evaporation test continued for 36 hours. After 36 hours, helium gas was used to pressurize the tank which allowed the LN2 to be drained back through the transfer line. To fully remove any residual liquid nitrogen, a vacuum pump was attached to the end vacuum transfer line. The pump ran for several hours to fully boil-off any remaining liquid. This method allowed for the removal of LN2 and also acted as a pre-cooling for the transfer line and the tank before the start of the LH2 experiments. Once the LN2 was fully removed and both the transfer line and the tank were fully evacuated, the tank was purged with helium gas and evacuated. The transfer of liquid hydrogen began. Hydrogen was transferred slowly into the tank allowing for a full cool-down with minimum transfer losses. The fill level was again

monitored with the liquid level sensor. Once the tank was full, data recording began. LH2 boil-off test was conducted for 36 hours to compare with 36 hours of LN2 boil-off.

## 3. Results and Discussion

### 3.1 LN2 and LH2 Boil-off Results

In Fig. 6 below, it can be seen that LN2 has an expected lower boil-off than LH2. This difference is due to the difference in enthalpies of vaporization and temperature differences of the two liquids. The average boil-off between hour 8, the start of pseudo-steady state, and hour 36, end of the test, was 0.04 SLPM for nitrogen and 0.43 SLPM for hydrogen.

There is a slight increasing trend in the data presented. Hydrogen flow at hour 8 was 0.42 but it did increase to 0.48 by hour 36. This increasing trend is perhaps from the increase in vacuum pressure resulting from the decreasing cold surface exposed to cryogenic temperatures as liquid level drops. The gas molecules, such as oxygen or other impurities, that initially stuck to the cold surface of the vapor shield and 5 L tank released as it slightly warmed resulting in lower insulation. Another possible explanation for this change in boil-off, as the liquid level drops, the top of the 5 L tank warms causing an increase

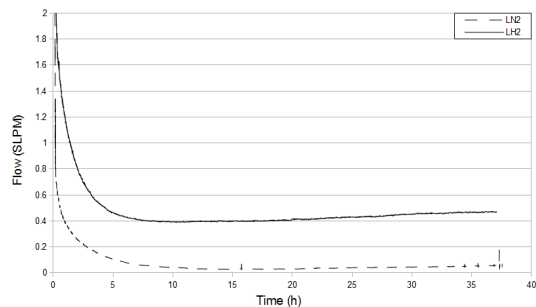


Fig. 6 Boil-off flow rate comparison over 36 hours

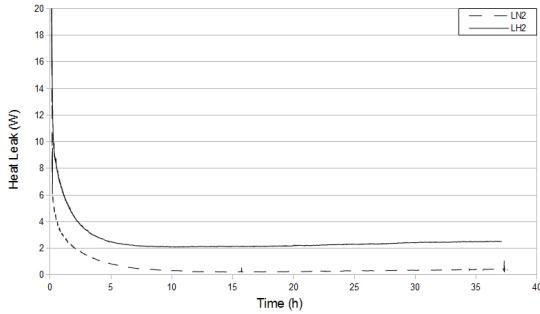


Fig. 7 Heat leak calculated from boil-off data

in heat load on the remaining liquid. There are three main sources of possible heat load in this system: conduction from the top flange down the G-10 filling tubes, convection heat transfer from residual molecules in the vacuumed annular space and radiation heat transferred from the outer tank to the inner tank. If one or more of these sources changed, there would be corresponding change in the heat load which is reflected in the boil-off.

From this flow data heat leak can be calculated from the thermo-physical properties of the desired fluid, as shown in Fig. 7. The average heat leak between hour 8 and hour 36 for LN2 was 0.31W and 2.28W for LH2. With this heat leak level and the associated boil-off, for 5L of LN2, it will take between 56 and 72 days to fully boil-off or 1.4~1.7% per day. For LH2 it will take between 6 and 7 days to fully boil-off or 14~16% per day.

### 3.2 Discussion

The target for this researched tank was 0.11 L/day boil-off or full boil-off in 66 days (1.5%/day) for liquid nitrogen and 0.57 L/day boil-off or full boil-off in 9 days (11.1%/day) for liquid hydrogen. The first LN2 target has been reached; the second LH2 target needs imp-

rovement.

Future experiments of both LN2 and LH2 can benefit from an improvement in vacuum level. The installation of the purchased TMP will help improve and lower the vacuum level in the chamber thereby reducing convective heat transfer. To help further improve the vacuum level, a bake-out preparation step could also be conducted. A bake-out should entail heating the tank to 100~120°C under nitrogen and then evacuated. This will fully drive off any moisture in the tank and it will also cause hydrogen trapped in the stainless steel to be released more easily thereby reducing off-gassing under vacuum.

Due to the slight drift in the data, future experiments should be conducted over longer periods of time. Longer time will allow for a better look at performance. This is a batch process so steady state will never be reached but a look at full boil-off of the 5 L will allow for a better conclusion of heat leak. Due to the very low flow rates seen, the experiment could also improve accuracy of low flow data with a higher resolution MFM.

More details about the design, fabrication, experiments, and performance test of a 5 L liquid hydrogen storage system were described in the reference<sup>13)</sup>.

## 4. Summary

A 5 L cryogenic storage tank was successfully designed, built and tested for this paper. Based on the design, thermal and structural analysis, the 5 L liquid hydrogen tank was fabricated and its performance tests have been carried out. The goal for this research was building a cryostat with 0.11 L/day boil-off or full boil-off in 66 days (1.5%/day) for liquid nitrogen and 0.57 L/day boil-off or full boil-off in 9 days (11.1%/day) for liquid hydrogen. The first LN2 target has been reached with a

boil-off rate of 1.4-1.7%/day. For LH2, the boil-off of 14.3-16.8%/day just missed the target boil-off of 11.1%/day. Future improvements in the system will allow the tank to fulfill the second goal. Continuing this research and conducting future experiments will lead to the improvement of storage technology for cryogenic liquids. In the future, these experiments may help to make hydrogen a liquid fuel of today rather than just a possibility of tomorrow.

## Acknowledgements

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