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## Performance and results of the LBNE 35 ton membrane cryostat prototype

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### Abstract

We report on the performance and commissioning of the first membrane cryostat to be used for scientific application. The Long Baseline Neutrino Experiment (LBNE) has designed and fabricated a membrane cryostat prototype in collaboration with Ishikawajima-Harima Heavy Industries Co., Ltd. (IHI). LBNE has designed and fabricated the supporting cryogenic system infrastructure and successfully commissioned and operated the first membrane cryostat. Original goals of the prototype are: to demonstrate the membrane cryostat technology in terms of thermal performance, feasibility for liquid argon and leak tightness; to demonstrate that we can remove all the impurities from the vessel and achieve the purity requirements in a membrane cryostat without evacuation; to demonstrate that we can achieve and maintain the purity requirements of the liquid argon using mol sieve and copper filters. The purity requirements of a large liquid argon detector such as LBNE are contaminants below 200 parts per trillion (ppt) oxygen equivalent. LBNE is planning the design and construction of a large liquid argon detector. This presentation will present requirements, design and construction of the LBNE 35 ton membrane cryostat prototype, and detail the commissioning and performance. The experience and results of this prototype are extremely important for the development of the LBNE detector.

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## 1. Introduction

The Long-Baseline Neutrino Experiment (LBNE) project envisions using membrane tank technology for a large liquid argon detector referred to as the Far Detector (FD) with the start of the construction in the 2020 time frame and operations in 2024 to do neutrino physics. The current detector configuration has a total Fiducial Mass of 34,000 tons to be located at the 4850L of the Homestake mine, in Lead, SD, USA, about 1.5 kilometer underground. It is being developed in a phased approach, where there are initially two cryostats of about 7,000 cubic meters of total volume each, followed by other two of about 15,000 cubic meter of volume each. Each cryostat will be instrumented with Time Projection Chambers (TPCs) and filled with liquid argon.

In order for the TPCs to operate properly and drift electrons with a lifetime greater than 1.4 millisecond (ms), the LBNE-FD requires extreme purity of the liquid argon inside the tank, at the level of 200 parts per trillion (ppt) of oxygen equivalent contamination. The empirical correlation between electron lifetime and parts of oxygen equivalent is outlined in equation 1:

$$\text{Lifetime [s]} = \frac{3 \times 10^{13} [\text{parts of Oxygen}]}{\text{Contaminant [parts of Oxygen]}} \quad (1)$$

In order to ensure that the membrane cryostat technology is suitable for this experiment, we have designed, built, and successfully operated the LBNE 35 ton prototype, a 29 cubic meter membrane cryostat. It is the first and only membrane cryostat for scientific purpose and available to scientists and the first on the US soil. Design and assembly were performed in collaboration with Ishikawajima-Harima Heavy Industries Co., Ltd. (IHI). Technical goals of this prototype are: to demonstrate the membrane cryostat technology, thermal performance, feasibility for liquid argon, leak tightness; to demonstrate that we can remove all the impurities from the vessel and achieve the purity requirements in a membrane cryostat without evacuation; to demonstrate that we can achieve and maintain the purity requirements of the liquid argon using molecular sieve and copper filters. These filters come from the LAPD experiment, a liquid argon R&D project that successfully reached 5.0 ms lifetime in a non evacuated cryostat [1]. The molecular sieve is type 4A from Sigma Aldrich; the copper filter is type CU-0226S from BASF.

Table 1 presents the list of the design parameters for the LBNE 35 ton membrane cryostat.

Two previous papers outline the full description of the membrane cryostat design and construction, in collaboration with IHI [2], and the cryogenic system design and installation by Fermilab/LBNE [3]. Fig. 1 (a) shows the 3-D drawing of the LBNE 35 ton membrane cryostat. Fig. 1 (b) shows the Process Flow Diagram.

Purity in liquid argon is measured by using double gridded ion chambers called Purity Monitors, described in [1]. Schematically, a Purity Monitor is composed of a cathode, cathode grid, a relatively long drift region, an anode grid, and an anode. This structure produces three regions; the cathode to cathode grid, the cathode grid to anode grid (the drift region), and the anode grid to anode. The cathode, a gold-plated aluminum plate, is illuminated by ultra-violet (UV) photons from a xenon flash lamp. These photons liberate electrons from the gold layer into the liquid argon. The electrons drift towards the cathode grid that is 1.8 cm from the cathode. The electric current from this drift between the cathode and the cathode grid is integrated in external electronics and represents the electron charge  $Q_c$  that was emitted from the cathode. These electrons continue to move through the long drift region towards the anode grid. Oxygen and water can capture individual electrons. Once the remaining electrons pass the anode grid, a current is developed on the anode, which like the cathode case, is integrated in external electronics. The ratio of the received anode charge  $Q_a$  and the emitted cathode charge  $Q_c$  gives a measure of the concentration of the electronegative impurities in the LAr. The electron lifetime,  $\tau$ , of an electron in liquid argon is defined from the formula 2:

$$Q_a = Q_c \times e^{-\tau/d} \quad (2)$$

Table 1. Design parameters for the LBNE 35 ton prototype.

Design parameter	Value
Cryostat volume	29.16 m <sup>3</sup>
Liquid argon total mass	38,600 kg
Inner dimensions of the cryostat	4.0 m (L) x 2.7 m (W) x 2.7 m (H) - flat plate to flat plate
Depth of the liquid argon	2.565 m (5% under plate A, total ullage 11%).
Insulation	0.4 m Polyurethane foam
Primary membrane	2.0 mm thick 304 corrugate stainless steel
Secondary barrier	0.1 mm fiberglass
Vapor barrier	1.2 mm thick carbon steel
Reinforced outer concrete layer	0.3 m thick
Liquid argon temperature	89 K +/- 1 K
Operating gas pressure	70 mBar
Vacuum	No vacuum
Design pressure	207 mBar
Leak tightness	1E-06 mBar*liter/s
Heat leak	< 13 W/m <sup>2</sup>
Duration	10 years
Thermal cycles	50 complete cycles (cool down and total warm up)
Design Codes	Fermilab ES&H Applicable parts of JGA RP-107-02 ACI 318

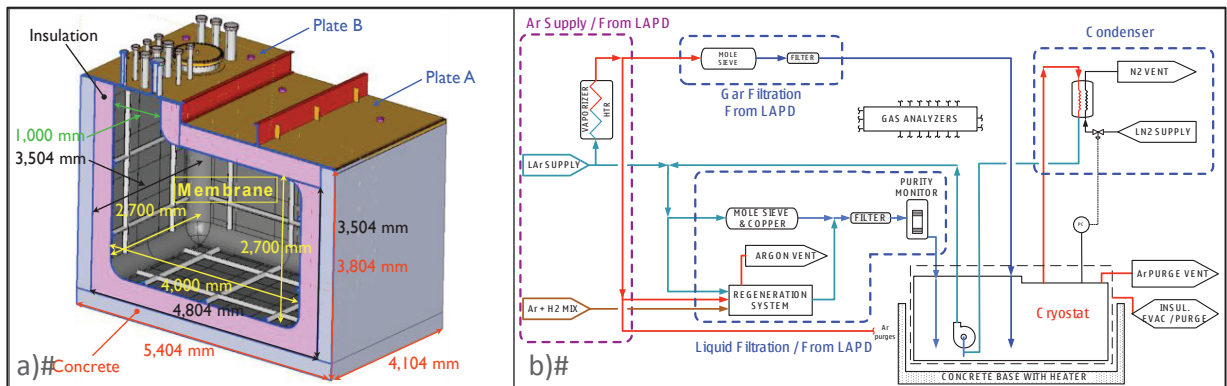


Fig. 1. (a) Membrane cryostat 3-D drawing; (b) Process Flow Diagram (PFD).

## 2. Performance and operations

The cryostat was initially filled with room temperature ambient air. The first step of the commissioning was the piston purge with unfiltered argon gas from a commercial 180 liter liquid argon dewar. The gas was injected at the bottom of the tank through a manifold that distributed it all across the floor and slowly rose to the top pushing the impurities out. The flow rate was 210 standard liter per minute (SLPM), which corresponds to a linear rate of rise of 1.2 meter per hour and a volume exchange every 2.4 hour, faster than the diffusion rate of air into argon. After the initial volume change the density difference in the tank is not significant anymore and the purge follows a model of perfect mixing. During these and subsequent processes, the gas is sampled from capillaries located at different

elevations underneath plate B and sent to gas analyzers to measure the concentration of the contaminants. Fig. 2 (a) shows the results of the piston purge: oxygen is reduced from 21% to 6.3 ppm, water from 550 ppm to 1.2 ppm, and nitrogen from 78% to 10.6 ppm. During this process the gaseous argon is vented out of the top of the tank. After the completion of this phase, before proceeding to the following one, we have purged the trapped volumes with cycles of pressurization/depressurization to force eventual residual impurities out.

The second step of the commissioning was the gas recirculation. The gaseous argon that comes out of the top of the tank is no longer vented, but sent to the purification system (water and oxygen filters) through a metal bellows recirculation pump, and then back to the bottom of the tank. It enters the tank through the same manifold used for the previous step. The process is similar to the piston purge: the gaseous argon enters from the bottom of the tank and pushes the impurities out from the top, but instead of being vented outside it is sent to the purification system, where the impurities are removed, and then back inside the tank. The gas recirculation flow rate was 135 SLPM, which corresponds to a volume exchange every 3.2 hours. Fig. 2 (b) shows the results of this process: oxygen is reduced from 49 ppm to 0.1 ppm, water from 10 ppm to 0.4 ppm, while nitrogen remained around the same value at 48 ppm. While we filter oxygen and water, we do not actively remove nitrogen. We slowly, but constantly, added make up argon gas to compensate the loss in the samplers. Any nitrogen reduction is therefore due to dilution of the existing amount into the small amount of fresh gas added to the system. Because of the density difference between liquid and gas phases (liquid argon is about 820 times denser than the gas) even 100 ppm of nitrogen in argon gas are not a problem for the final purity: they will be about 0.1 ppm in liquid argon. Oxygen and water however need to be reduced to less than 200 ppt and are therefore actively removed in the copper beads and molecular sieve.

The third step was the cool down and filling. We designed a vapor phase cool down scheme involving spray nozzles to follow IHI's cool down guidelines: the membrane cryostat had to be cooled down at less than 10-15 kelvin per hour [4]. We have developed and constructed a system that sprays liquid argon from a center nozzle and warm gaseous argon from two angled nozzles pointing at the flow of liquid. The liquid is then atomized into a fine flat spray of cold argon gas containing liquid droplets that removed heat from the tank by evaporation before impinging on any cryostat metal surface. Additional gas only nozzles injected room temperature gas into the tank to provide momentum to keep the tank mixed. This solution was driven by the need to follow IHI's guideline for the cool down and the need to avoid spraying cold liquid or gas over the TPCs that will be installed in the LBNE Far Detector cryostat, but also inside this prototype during the phase 2 tests. CFD analysis was used to determine the size and number of sprayers and nozzles. Four liquid spray nozzles were installed with a design capacity of 0.9 LPM of liquid argon and 235 SLPM of room temperature gaseous argon. Three gas only nozzles were also installed with a combined design capacity of 210 SLPM of room temperature argon gas. Fig. 2 (c) shows the temperature of the membrane cryostat during cool down, well within IHI's specifications, as measured by temperature sensors glued over the membrane panels inside the tank. Immediately after reaching the liquid argon temperature, we started filling the tank with liquid argon coming from the LAPD tank, which is a little smaller than the 35 ton prototype, reaching 70% full, or about 19,000 liter.

The fourth and last step is the operations: to continuously purify the liquid and measure the electron lifetime. After the initial fill, one of the two submerged liquid argon pumps started to send liquid argon from the cryostat to the cryogenic purification system to remove oxygen and water from the bulk of the liquid argon. The liquid argon flow rate was set at 35 SLPM, which corresponds to a volume exchange every 9.2 hour. It is more difficult to achieve the purity in a partially filled tank, because of the higher warm surface that is outgassing. Fig. 2 (d) shows the electron lifetime as a function of the volume changes. It took about 5.5 volume changes to reach 1.0 ms, which corresponds to about 300 ppt of oxygen contamination. It took about 14 volume changes to reach the LBNE requirement of 1.4 ms, which corresponds to about 200 ppt of oxygen contamination. After about 45 volume changes we added about 7,500 liter of liquid argon from the decommissioned DZERO calorimeter. During the fill, the submerged liquid argon pumps were off and the purity dropped down to 0.35 ms, quickly rising back up once the filtration was restarted soon after the completion of the fill. At that point it took only 10 volume exchanges to reach 3.0 ms lifetime, which corresponds to about 100 ppt oxygen contamination.

We have measured the static heat leak of the cryostat to be less than 1.5 kW total, with the cryostat isolated from the piping system and the pumps off. The walls, floor and plate A have been estimated by IHI during the design phase at the order of 1.3 kW, with the balance coming from plate B, which only has radiation shields, and not polyurethane insulation underneath.

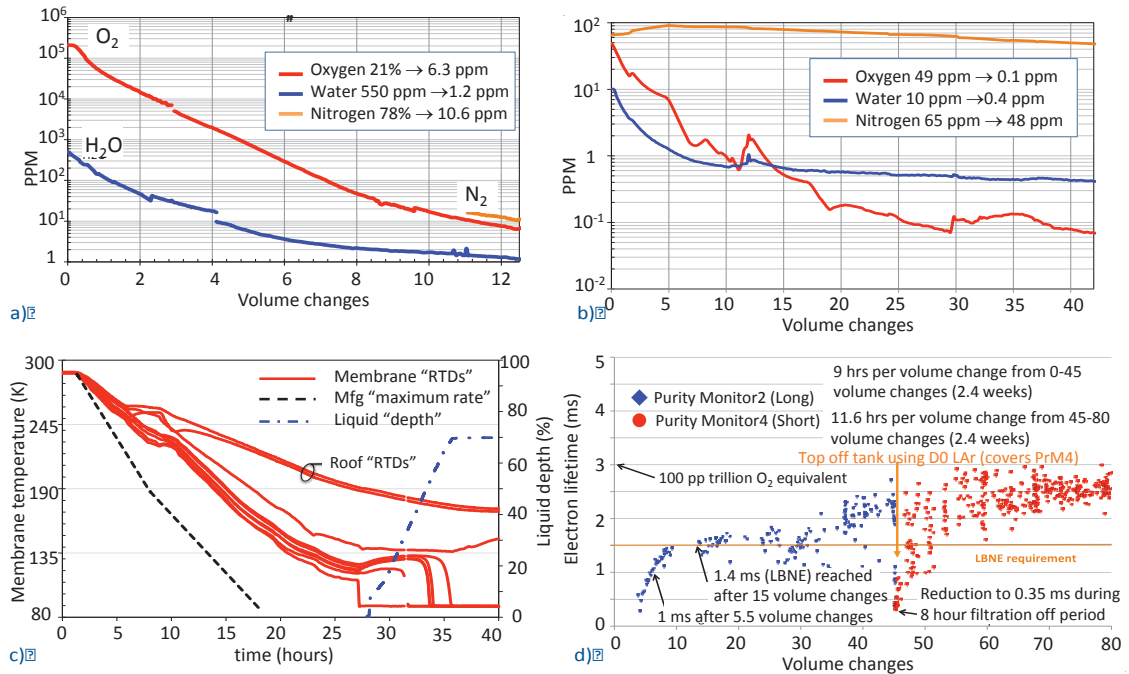


Fig. 2. (a) Step one: piston purge; (b) Step two: gas recirculation; (c) Step three: cool down and fill; (d) Step four: operations.

### 3. Lessons learned

During the gas recirculation we have found a leak in one of the seals of the vacuum relief valve. The contamination was estimated as one standard cubic centimeter per minute (sccm). We were able to reduce it down to 0.06 sccm by bagging the relief with a gaseous argon purge.

During cold operations (at around 200 K), we have developed an argon gas leak from the argon cryo-piping into the vacuum jacket sleeve around it and identified the seal of the dielectric breaks as the source. While the dielectric breaks on the cryo-piping (an LBNE requirement to electrically isolate the cryostat from the remaining of the building) are leak tight at room temperature, they are not at cryogenic temperatures and we have addressed the issue by actively pumping the vacuum jacket sleeve around the piping to maintain a good insulating vacuum throughout the whole process.

The Purity Monitors were found to be very sensitive to microphonics. They are rigidly connected to plate B, which supports also the circulation pumps. We are looking into possibly decoupling the purity monitors and or the pumps from plate B or stiffening plate B itself to reduce the transmission of the sound waves.

One of the two submersible liquid argon pumps, pump B, failed after about 770 hour, probably because of a bearing failure. Fig. 3 (a) shows a filter of pumps A and B. It is the filter located on a small line after the discharge, where a tiny flow of liquid argon is sent to cool down and lubricate the bearings and the motor. Fig. 3 (b) shows a zoom in view of the filter of pump B. Fig. 3 (c) shows a detail of the filter of pump A with some molecular sieve, yellowish on the left, and copper beads, blackish on the right. We are still investigating the issue, but we believe that molecular sieve crumbles have been transported back along the long transfer lines that connect the 35 ton membrane cryostat to the purification system by the gaseous argon bubbles generated when the pumps were not running. The crumbles that were carried back clogged the filters wearing down the bearings. A surface blackish substrate is evident on the filter of pump B, the one that failed. We think that that is bearings material that have deteriorated because of lack of appropriate coolant and lubricant caused by the clogged filter. The investigation continues and more details will be presented at a later time.

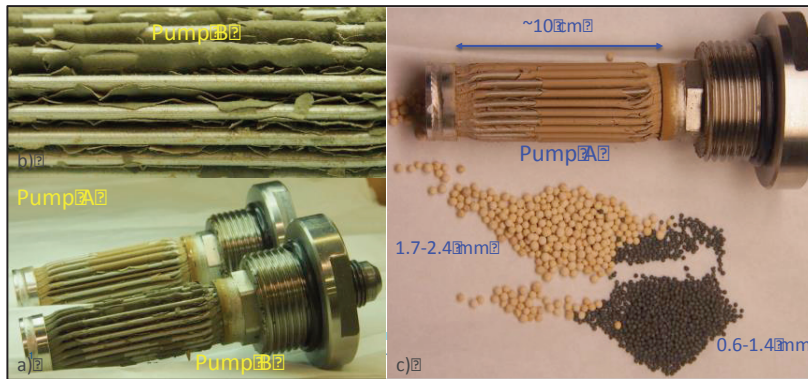


Fig. 3. (a) Filters of LAr pumps A and B; (b) details of filter of pump B; (c) filter of LAr pump A with molecular sieve and copper beads.

#### 4. Conclusion

In 2012 we have completed the installation of the LBNE 35 ton membrane cryostat in collaboration with IHI. In 2013 we have completed the installation of its associated cryogenic system, including the connections to the existing liquid argon filtration and purification system from LAPD. We have recently successfully commissioned and operated the first membrane cryostat for scientific application, and the first on US soil, and its associated cryogenic system (LBNE 35 ton prototype phase 1). We have measured 2.5-3.0 ms electron lifetime, which equals about 100 ppt Oxygen equivalent contamination, exceeding the LBNE design requirements of at least 1.4 ms lifetime, or less than 200 ppt Oxygen equivalent contamination. We have demonstrated the membrane cryostat technology, in terms of thermal performance, feasibility for liquid argon, and leak tightness. We have demonstrated that we can achieve and maintain the purity requirements in a membrane cryostat without evacuation, and using only a slow gaseous argon purge. We have demonstrated that we can achieve and maintain the purity requirements for the liquid argon during filling, purification, and maintenance mode using molecular sieve and copper beads.

#### 5. Future steps

A second set of tests, known as the LBNE 35 ton prototype phase 2, is under way with the installation of reduced-size scale TPCs inside the membrane cryostat and the addition of muon counters on the top and sides of the tank. The goal for this phase is to validate the design of nearly all TPC components (at a small scale) and take data on purity, electronics, noise, ion space distribution. The plan is to start the installation of the TPC at the beginning of the fall and the data taking by the spring of 2015. The completion of these tests will represent an important milestone for the LBNE project, as this was thought to be the only prototype before the construction of the Far Detector. There are no scheduled activities after the completion of phase 2. We envision a potential phase 3 where the prototype is available to scientists and researchers other than LBNE.

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