IOP Conf. Series: Materials Science and Engineering **1344** (2026) 012097 doi:10.1088/1757-899X/1344/1/012097

Simulation experiment of vacuum insulation deterioration in liquid hydrogen tank due to minute air leaks

Suguru Takada[1*], Shinji Hamaguchi[1], Shigeyuki Takami[1], Masakazu Nozawa[2] and Hiroaki Kobayashi[3]

[1] National Institute for Fusion Science, National Institutes of Natural Sciences, Toki, Gifu, Japan
[2] KOSEN of King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand
[3] Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency, Sagamihara, Kanagawa, Japan

*E-mail: takada.suguru@nifs.ac.jp

Abstract. An experimental study on the safety of a liquid hydrogen (LH_2) storage tank and transfer tube equipped with vacuum insulation has been carried out. A small-scale setup was used to simulate minute air leakage from the outside, caused by deterioration of the O-ring in the vacuum seal-off valve. This experiment investigated the increase in vacuum pressure when air was introduced into a vacuum chamber submerged in LH_2, leading to the deposition of solid air components on the inner walls. A leak rate of approximately 10-3 Pa m^3/s was introduced, revealing a slow pressure increasing to around 1 Pa by a long time constant. Additionally, an alternative method involving the leakage of carbon dioxide (CO_2) into a test chamber submerged in liquid nitrogen (LN_2) was examined to facilitate repeated experiments under safer conditions. Opportunities for LH_2 experiments are limited due to strict safety requirements. In contrast, the pressure increase observed in the LN_2 experiments was greater than that in the LH_2 experiments.

1. Background

A cryogenic storage tank or transfer tube is equipped with a vacuum insulation layer to minimize heat transfer from the surroundings. Most vacuum insulation layers are sealed with valves using rubber O-rings. Thus, operators have sometimes experienced vacuum loss caused by aging-related deterioration of rubber O-rings during long-term operations. The deterioration of rubber O-rings is difficult to predict, as aging depends strongly on environmental conditions. Therefore, operators of cryogenic systems must detect early signs of abnormality.

However, LH_2 and liquid helium have the ability to condense trace amounts of leaked air due to their extremely low boiling points. Therefore, it is expected that the vacuum pressure remains sufficiently low to maintain effective insulation through the cryo-pumping effect, even in the presence of minute air leaks caused by a deteriorated sealing O-ring. The questions have arisen how much leak rate can be allowable in LH_2 storage tank and how vacuum degradation progresses.

Previous studies have simulated severe accident scenarios, such as sudden vacuum failure, to evaluate system robustness [1–3]. In contrast, the present experimental study aims to investigate the process of vacuum degradation caused by minute leaks in LH_2 vessels or transfer tubes. This research addresses a gap in current knowledge regarding safety risks associated with aging-related deterioration during long-term operation.

2. Experimental setup

2.1 Air leak test setup in LH_2

Figure 1 shows a schematic drawing of the experiment. The vacuum tube was immersed in liquid hydrogen (LH_2) within a glass dewar. A room-temperature-controlled tube was configured to introduce leaked gas into the vacuum tube. The inner tube simulated the exterior of a cryostat, and the wall facing the LH_2 simulated the inner wall of the cryostat's LH_2 vessel. Two manganin wires, each with a resistance of approximately 40 ohms, were wrapped around a copper tube with an outer diameter of 6.35 mm to maintain room temperature. The input power to each heater was measured separately. Additionally, the inner tube was covered with 20 layers of multi-layer insulation over the heater wires. The liquid level was monitored by visual inspection and several PtCo resistance thermometers. Throughout all experiments, the liquid level remained between 160 mm and 300 mm from the bottom of the chamber. The vacuum tube had an inner diameter of 39.4 mm, an outer diameter of 42.7 mm, and a length of 600 mm, fitted with an ICF70 flange. The inner tube was 550 mm in length. A stainless-steel tube (SUS304) of BA class (bright annealed) was selected, with roughness of surface comparable to that of a standard cryogenic liquid transfer tube. The average surface roughness was Ra < 0.8 μm, and the maximum peak-to-valley height was Ry < 3.0 μm.

The leaked air was supplied from an 8-liter buffer tank filled with dried air sourced from a high-pressure cylinder. Although impurity gases such as helium and hydrogen, which do not solidify at 20 K, may have been present at concentrations of several tens of ppm, they were not measured in the present experiment. The temperature was maintained at room temperature, and the leak rate was controlled using a low-pressure buffer tank (several tens of kPa) and two low-conductance valves in series, achieving a leak rate on the order of 10^{-3} Pa m^3/s. Pressure in the vacuum vessel (P1) was measured using a full-range gauge combining a Pirani and a cold cathode sensor. The leak rate was calculated from measurements of pressure (P2), temperature, and volume of the buffer tank using a diaphragm pressure gauge. After applying temperature correction, the variation in leak rate was within a few percent. The pressure measurement at P1 was limited in accuracy, with only two significant digits and an error margin of ± 15 % above 10 Pa and ± 50 % below 10 Pa. In contrast, the measurements of P2, temperature, and volume used for leak rate calculation were more precise, with a combined error below 1 %.

2.2 Procedure of the experiment

To ensure complete drying of the test chamber interior, all connected components were evacuated five times using a scroll pump. Dry artificial air from a high-pressure cylinder was introduced while heating the system to approximately 343 K. After sealing the gate valve, the test chamber

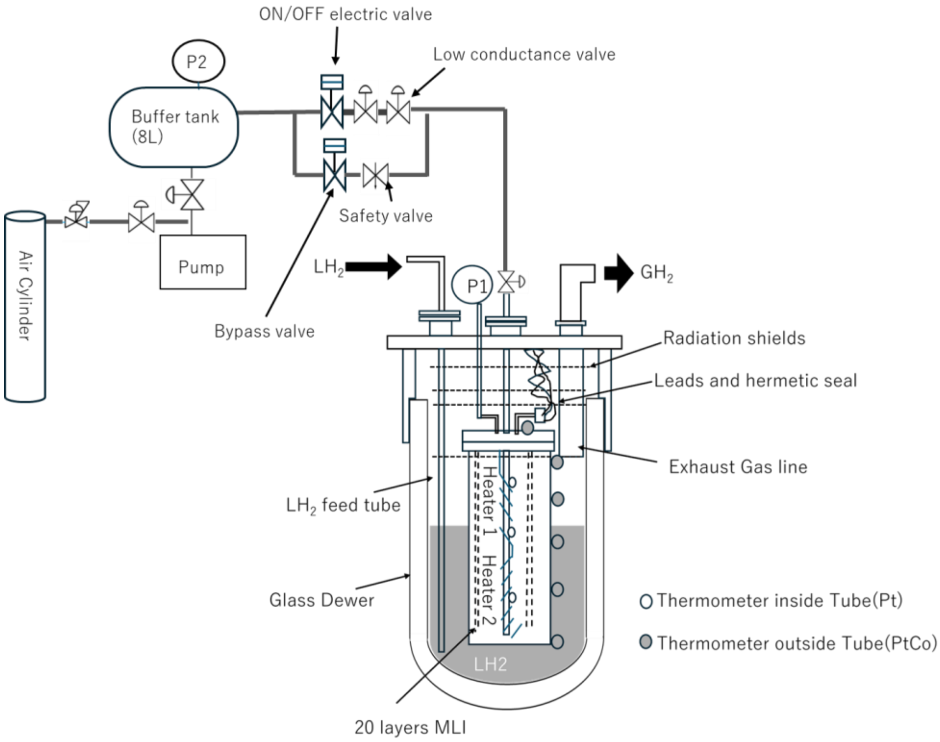

Figure 1. Schematic illustration of minute air leak test in LH_2

was attached to the glass chamber. With all valves open, the system was evacuated to below 5 Pa using a scroll pump. The bypass valve and electric ON/OFF valve were then closed. Under these conditions, the system's build-up rate was evaluated by monitoring the pressure increase at sensor P1. The observed rate, approximately 10^{-6} Pa m^3/s, was likely caused by minor leakage through the O-rings in the quick couplings or by residual condensed gas on the internal surfaces of the test chamber and associated piping. Based on this pressure increase, the estimated leak rate of the entire system exceeded the nominal performance of the O-ring quick couplings by more than an order of magnitude. This discrepancy is attributable to the use of 15 rubber O-ring flanges in the room-temperature sections of the system, in contrast to the metal flanges employed in the cryogenic regions.

Subsequently, LH_2 was injected into the glass dewar. The ICF70 flange was connected via a 6.35 mm pipe wrapped with a heater, and the upper surface of the test chamber was maintained above 220 K. Additionally, the temperature control pipe inside the chamber was regulated by a heater to ensure it consistently exceeded 270 K. An 8-liter buffer tank was filled with approximately several tens of kPa of dried air. After the LH_2 transfer, residual gas condensed, resulting in a pressure drop to below 0.05 Pa. At this stage, the ON/OFF electric valve was opened to initiate the leakage process. The leak rate was determined by both the pressure in the 8-liter buffer tank and the opening angle of the two hand valves. The leak rate was calculated from the pressure decay measured at P2.

2.3 Alternative test using liquid nitrogen and carbon dioxide.

Experiments using liquid hydrogen (LH_2) require strict safety and rather high operational costs, which significantly limit both accessibility and frequency of testing. In addition, the number of available test facilities capable of handling LH_2 is limited. Therefore, as an alternative test, the comparative experiments were conducted using liquid nitrogen (LN_2) and carbon dioxide (CO_2). As shown in Figure 2, the saturation pressure of CO_2 at approximately 77 K (the temperature of LN_2) is sufficiently low to enable vacuum insulation, which is similar to the saturation pressures of nitrogen and oxygen at around 20 K (the temperature of LH_2) [4, 5]. Moreover, the condensation rates and the capture coefficients of these gases are also similar [6]. In general, the cryo-pumping effect is characterized by the theoretical maximum pumping speed and the capture coefficient. They can be described by the following Equation (1) and Equation (2)

$$S = CS_{theo} \quad (1)$$

$$S_{theo} = \sqrt{\frac{RT_g}{2\pi M}}\left(1 - \frac{P_s}{P_c}\right) \quad (2)$$

C is the capture coefficient, S_{theo} is the theoretical maximum pumping speed, R is the gas constant, T_g is the temperature of leak gas, M is the weight of molecules, P_s is the vapor pressure of condensate at temperature of cryo-surface, and P_c is the pressure of vacuum chamber. The pumping speeds per area are in the order of 100 Pa m^3/s [7, 8]. Another study reported that, below 1 Pa, the pumping speed with a stainless steel cryo-surface is much lower than with a copper surface [9].

Considering the increase in heat transfer due to vacuum deterioration, the mean free path is also an important factor. For instance, at room temperature and 1 Pa pressure, the mean free path of CO_2 is estimated to be 3.9 mm, while that of N_2 is 6.6 mm. Although there is a difference, it is not significantly large. This LN_2 experiment was conducted using the same setup as shown in Figure 1. In the case of LN_2 experiment, it was accumulated in an open container with a height of 300 mm. As a result, the temperature gradient above the liquid surface was steep, and the condensation area was almost equivalent to the area submerged in the liquid. This aspect differs from the LH_2 experiment. In contrast, during the LH_2 experiment, the temperature gradient was more gradual, with the region 200 mm above the liquid surface maintaining approximately 50 K. The estimation of the condensation area in this setup was not clear in the case of the LH_2 experiment.

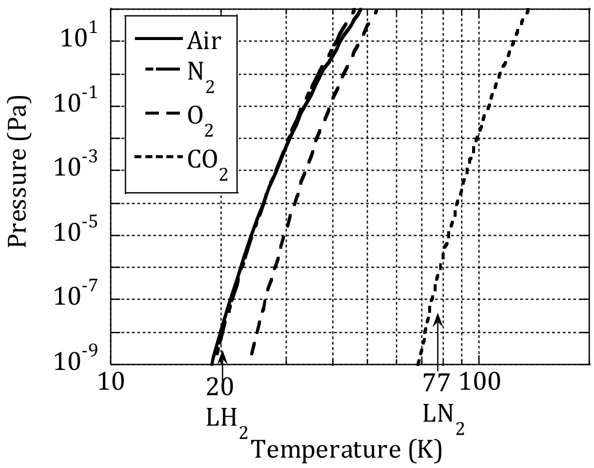

Figure 2. Saturation curve of nitrogen, oxygen and carbon dioxide replotted by *REFPROP* [5]

IOP Conf. Series: Materials Science and Engineering **1344** (2026) 012097 doi:10.1088/1757-899X/1344/1/012097

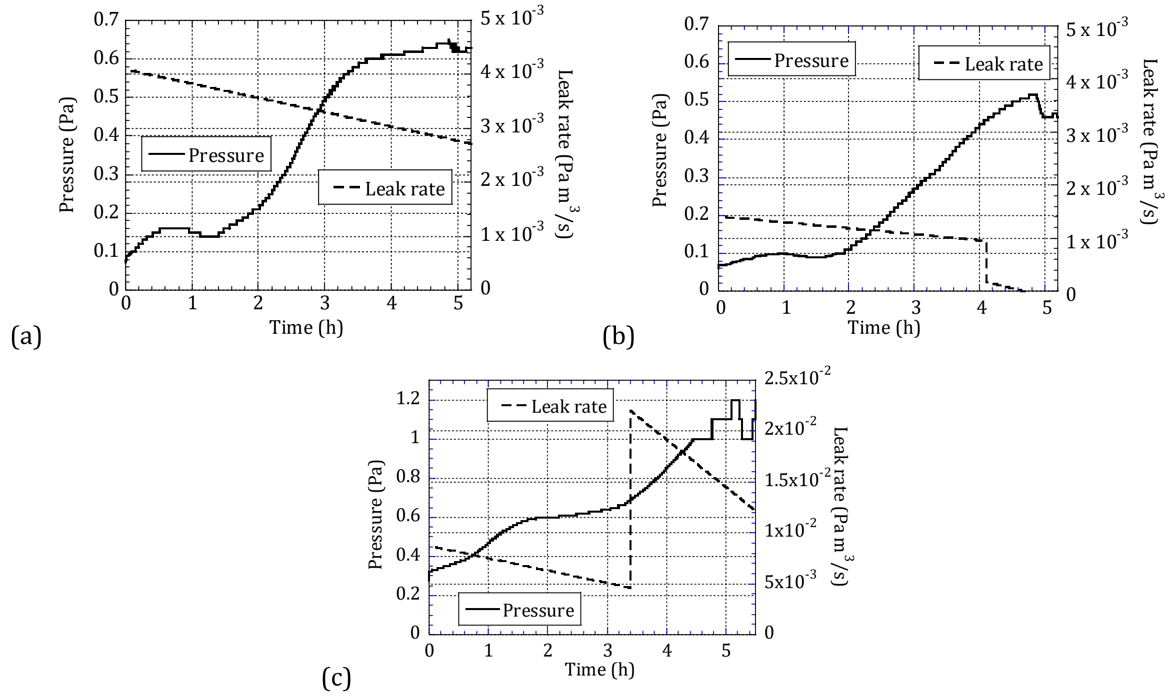

Figure 3. Time variation of pressure growth P1 in the test chamber and the leak rate calculated by the measurement of pressure decrease in the buffer tank P2 in LH_2 while minute air leakage. (a) 4.0 - 2.7 x 10^{-3} Pa m^3/s, (b) 1.4 - 0.9 x 10^{-3} Pa m^3/s. Gate valve was shut after 4 hours, (c) 8.6 - 4.4 x 10^{-3} → 2.2 - 1.2 x 10^{-2} Pa m^3/s, filling the buffer tank in a minute after 3.5 hours.

3. Results and discussion

LH_2 experiments were conducted in three cases as shown in Figure 3 (a)-(c). Though the leak rate has been decreased accompanying with the pressure decay of the buffer tank, the pressure of vacuum tube has kept growth in all cases. The liquid level has decreased linearly from about 280 to 160 mm. The heat input of the leak tube inside the vacuum tube were growth accompanying with the vacuum pressure in the range of several watts. The influence of heat penetration due to gas condensation and heater to maintain room temperature of leak tube was rather small compared with the heat penetration from this systematics. The heat flux across the wall of vacuum tube is hard to estimate because of the vertical temperature distribution of the wall. However, assuming roughly that the heat input of “Heater 2” in Figure 2 is distributed uniformly to the half level of the vacuum tube, the temperature difference in the thin stainless-steel wall should be less than 1 K.

In the case of Figure 3 (a), a bump around 0.1 Pa was observed, gradually increasing over a 5-hour period. In the case of rather small leak rate shown in Figure 3 (b), a bump around 0.1 Pa slightly below the case of Figure 3 (a) also were observed slightly slower. After approximately 4 hours, the valve was closed to eliminate the leak, but the pressure continued to rise and began to decrease after more than 30 minutes. It is indicated that it is a remarkably long time constant. Regarding the observed bump, it is hypothesized that multiple phenomena with combined time constants contribute to this behavior, including gas adsorption onto the multilayer insulation (MLI) and sublimation from solid materials. These processes are likely influenced by variations in the temperature gradient established within the 20-layer MLI structure. The observed bumps of Figure 3 (a)-(c) have shown a correlation wherein an increase in the leak rate results in a higher peak value and a more gradual decline. Furthermore, despite the sudden rise of the leak rate, the vacuum pressure demonstrated a continuous increase slowly over time in the case of Figure 3 (c). Although this study suggests the importance of conducting longer-duration experiments, the current experimental constraints have limited test durations to under six hours.

The pumping speed of the cryopump, calculated based on the measured leak rate, was found to be significantly lower than the values commonly reported in previous cryopump studies [6–8]. For instance, in the case shown in Figure 3 (a), assuming the cryo-surface height corresponds to the liquid level, the pumping speed per unit area can be calculated to be approximately 1.1×10^{-6} to 7.4×10^{-7} m^3/m^2 s on NTP. Hord [9] reported that the pumping speed of a stainless-steel tube is considerably lower than that of a copper plate at pressures below approximately 1 Pa. However, the observed differences between the copper plate and the stainless-steel tube such as variations in thermal resistance and the availability of condensation nucleation sites cannot be fully explained by these factors alone. The thermal resistance of stainless steel can make temperature difference of only about 0.1 K under the heat penetration from the temperature-controlled leak tube. By referencing Equations (1) and (2) with the saturation curve of air, it becomes evident that the temperature gradient between the inner and outer surfaces of the stainless-steel pipe and the thermal resistance in condensate of air is insufficient to explain the observed low exhaust velocity. These equations implied that the condensation surface temperature would need to rise to approximately 35 K. It is an unrealistically high value. In present experiments, leakage air may contain non-condensable gas at 20 K. On the other hand, when 10 ppm of non-condensable gas contents were assumed, this discrepancy can be explained roughly. However, these strange bumps and slow reactions cannot be fully explained. More precise experiments are needed.

The experiments on LN_2 environment with CO_2 leaks were also conducted in a similar setup. The open container for LN_2 was used. Thus, the liquid level kept between 285 and 275 mm because the refilling is easy. Figure 4 shows typical experimental data. The leak rate was similar to the case of Figure 3 (a). The bump was not seen in the pressure growth. The vacuum pressure has growth earlier and higher compared with the LH_2 experiments.

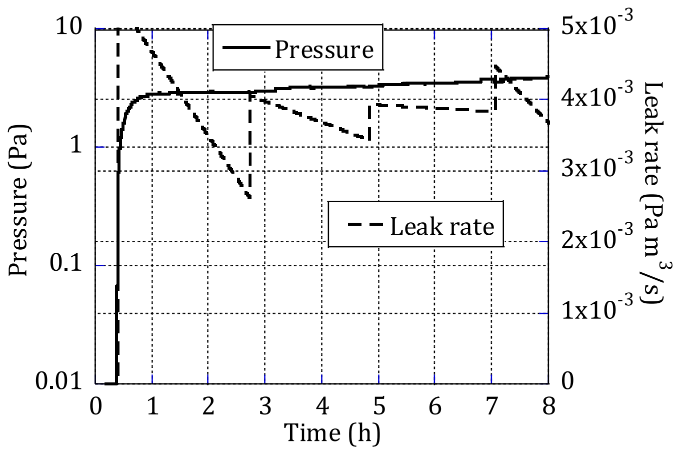

Figure 4. Typical time variation of pressure growth P1 in LN_2 with the leak of CO_2. To maintain the leak rate within a specified range over an extended period, gas was replenished into the buffer tank several times.

Considering the presence of gases that do not condense at 77 K, which must be contained in CO_2 in the experiments, the vacuum pressure should continue to increase. However, the vacuum pressure sometimes remains the same for over 1 hour. Figure 5 shows the relationship between this value and the leak rate. In Figure 5, the condition is referred to as "quasi-steady" when the data remains constant for over 30 minutes, and as "steady" when it remains constant for over 1 hour. The pressure gauge has only 2 digits, so it is hard to distinguish the steady state. The minute leak rate on the order of 10^{-4} Pa m^3/s has raised the vacuum pressure slightly below 1 Pa. It is indicated for cryogen tank that there is a region where it is difficult to predict the presence of a leak from pressure measurements during daily inspections.

And these experiments have been estimated in the range of "the free molecular flow" region and "the transition" region because the heat transfer coefficient has positive relation on the pressure as shown in Figure 6. The heat transfer coefficient was calculated based on the wet level, the heat input for the leak tube and the temperature difference between leak tube and the saturate temperature of LN_2. This calculation is not precise but the temperature gradient above the liquid surface was steep in the experimental setup for LN_2 so that it is enough to distinguish the flow regime. When the vacuum pressure is within the transition region, thermal conduction exhibits a positive correlation with pressure. Consequently, temperature difference in the frost formed by condensate may degrade the performance of the cryopump. Such thermal feedback could lead to a sudden pressure increase upon frost evaporation. Therefore, further experiments are necessary to quantitatively verify the conditions under which this feedback mechanism accelerates the rise in vacuum pressure against rather higher leakage rate.

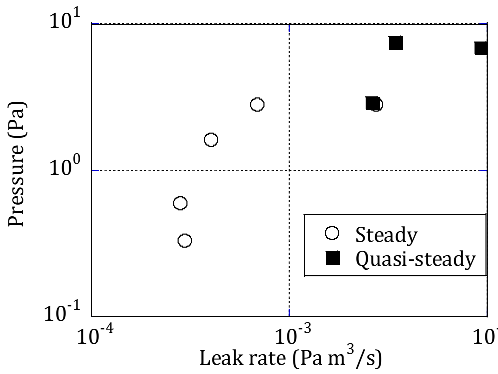

Figure 5. The pseudo- steady pressure on leak rate

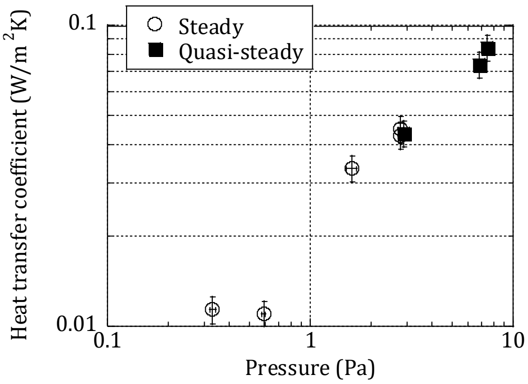

Figure 6. the heat transfer coefficient on the pressure of the vacuum tube calculated by the heat input and the temperature difference between leak tube and LN_2 77 K

4. Summary

A simulation experiment was conducted to know the process of the deterioration of vacuum insulation in a LH_2 tank when minute air leaks due to aging of an O-ring or other factors. We conducted a small-scale experiment using an inside-out inverted configuration of a vacuum insulation of LH_2 tank. After immersing a vacuum chamber, equipped with a temperature-controlled leak tube at room temperature, into LH_2, artificial air at an order of 10^{-3} Pa m^3/s was injected, and the resulting pressure variations were measured. The findings of the experiment revealed the following: In about 5 hours experiment with minute leak in the order of 10^{-3} Pa m^3/s, the pressure kept increasing gradually below 1 Pa. The cryo-pumping speed was extremely slow on inner wall. This may be explained mainly by non-condensable gas content at 20 K of about 10 ppm in Air. However, the curves of strange bump and slow reaction cannot be fully explained by the non-condensable gas. More precise experiments are needed.

To verify whether an alternative experiment could replicate air leakage into a vacuum layer at LH_2 temperature, we conducted a test by injecting CO_2 into a vacuum layer at LN_2 temperature. The CO_2 with LN_2 experiment led to faster and more pronounced pressure increases. This may suggest that the experiment using LN_2 and CO_2 could serve as a conservative indicator for safety assessments of LH_2 storage with air leakage.

Acknowledgments
This work was funded by a grant (No. MIS014) of National Institute for Fusion Science from the Ministry of Education, Culture, Sports, Science and Technology (MEXT) of Japan. This experiment was supported by staffs of JAXA Noshiro rocket test center at Akita in Japan. We also thank Mr. Hiroki Noguchi and Hiroyuki Tanoue (NIFS) for the help in development of the control system. We would like to thank all supporters of this project.

References

[1] Bosque E. S., Dhuley R.C. and Van Sciver S. W., 2014, *AIP Conference Proceedings* **1573**, 260
[2] Dhuley R.C., Bosque E. S. and Van Sciver S. W., 2014, *AIP conference proceedings* **1573**, 626
[3] Weber C., Henriques A., Zoller C. and Grohmann S., 2017, *IOP Conf. Series: Materials Science and Engineering* **278,** 012169
[4] Day C., Basics, and applications of cryopumps, 2015, *CAS - CERN Accelerator School: Vacuum in Accelerators,* 241-274
[5] *mini-REFPROP* ver. 10.0 by National Institute of Standards and Technology
[6] Dawson J. P., Haygood J. D., and Collins J. A.,1964, *Adv. Cryo. Eng.* **9**, 443-449
[7] Wang E.S. J., Collins J. A., and Haygood J.D., 1963, *Adv. Cryo. Eng.* **8**, 73-84
[8] Dawson J. P., Haygood J. D., 1965, *Cryogenics* **5**, Issue 2, 57-67
[9] Hord J., 1966, *Cryogenics* **6**, Issue 5,285-293