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Experimental study of moisture uptake of polyurethane foam subjected to a heat sink below 30 K

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ABSTRACT

Rigid closed-cell foam is widely used to thermally insulate liquid hydrogen and oxygen tanks of space launch vehicles due to its lightweight, mechanical strength and thermal-insulating performance. Up to now, little information is available on the intrusion of moisture into the foam that subjects one side to liquid hydrogen temperatures and the other side to room temperatures and high relative humidity. A novel cryogenic moisture uptake apparatus has been designed and fabricated to measure the moisture uptake into the polyurethane foam. For safety and convenience, two identical single-stage pulse tube cryocoolers instead of liquid hydrogen are used to cool one side of the foam specimen to the lowest temperature of 26 K. Total of eight specimens in three groups, according to whether there is a butt-joint or weathering period, are tested respectively for both 5 h and 9 h. The additional weight due to moisture uptake of the foam for the 26 K cases is compared to previous measurements at 79 K. The results are instructive for the applications of foam to the insulation of liquid hydrogen tanks in space launch vehicles.

1. Introduction

It is a must to provide good thermal insulation for the liquid hydrogen (LH_2) and liquid oxygen (LO_2) tanks on the space launch vehicles to avoid the large boil-off losses. The launch vehicles of CZ-3A and CZ-3B Serials in China [1] use polyurethane (PU) foam with CFC-11 as the blowing agent, while the ARIANDE 5 launcher uses the Polyvinyl Chloride (PVC). The polymethacrylimide (PMA, also known under the trade name Rohacell) foam was reported [2] for the potential usage in the high-speed reusable aircraft with comparable insulation performance to PU foam. The applications and properties of the PU rigid foam is introduced in Ref. [3].

The thermal properties of this foam at different temperatures are definitely the main concern, and have been widely investigated theoretically and experimentally in the past decades. It is recognized that heat transfer through the foam insulation occurs by conduction through the solid matrix and gas as well as by radiation through the whole medium [4,5]. Several models have been developed to predict the effective thermal conductivity, including the geometrical cell model by Placido et al. [4], and the analytical model by Tseng et al. [6]. In the experimental aspect, the thermal diffusivity of rigid PU foams blown with different hydrocarbons was measured by Prociak et al. [7] for calculating the effective thermal conductivity. Tseng et al. [6] tested the conductivity of PU foam with R-141b as the blowing agent in the temperature range from 20 to 300 K based on a two-stage GM cryocooler. The results indicated that the thermal conductivity of the PU foam can be reduced by as much as 70% by evacuating the gases in the foam cells. The thermal conductivity of PU foam designed for ARIANE 5 Launcher is experimentally determined by Fischer et al. [8] at 80 K and 20 K by using the LN_2 and LH_2, respectively. Recently, Barrios et al. [5,9] compared the thermal conductivity of foam for four cases: "as received", evacuated, conditioned and helium intrusion in the temperature range from 30 K to 300 K. The results verified that the residual gas inside the foam has a predominant effect. The conditioning process with one side at 78 K, which incurs moisture absorption, has little effect on the effective conductivity at the temperatures over 80 K.

In fact, when the foam is exposed to a high-humidity environment and an applied large temperature gradient for a period, it seems to be inevitable that water penetrates into the foam by vapor condensation. As a result, it may degrade the thermal performance and significantly increase the undesired weight. Therefore, it is necessary to determine the amount of water/ice taken into the foam insulation under practical cryogenic conditions of the space vehicles. Unfortunately, less attention has been paid on the effects of moisture uptake and condensation, in particular at cryogenic temperatures. The weight-based amount of water can be surprisingly high, approaching as much as ten times the dry sample

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reported for the expanded bead polystyrene foam (EPS) under near-ambient conditions [10]. Martyn et al. [11] reported the rate of water uptake for the aged PU foam with commercial formulations for nearly one month test period, under the conditions of 273 K on one side and 323 K and 75% RH on the other side. They found that the water content increases linearly with time and the final weight increases over twofold. At the cryogenic conditions, the moisture uptake will be even serious as the cryo-pumping effect is strengthened. Fesmire et al. [12] experimentally verified that the potential added lift-off weight is in the range of 1134–1633 kg with the mass of foam of 2200 kg on the space shuttle external cryogenic tank, for an on-time launch and substantially more weight penalty for a 24-h scrub turnaround and launch. The present authors have built a cryogenic apparatus for the measurement of the moisture uptake of the rigid PU foam at LN_2 temperatures [13]. We explored the effects of the surface thermal protection layer, the thickness, exposed time, cracks and the material density.

The moisture uptake processes are rich in physics. An impressive progress on water uptake mechanism was made recently by Vanderlaan et al. [14], who firstly obtained the 3D images of water content in the conditioned foam samples using the method of MRI. Here, "conditioned" means the sample was launch pad conditioned in a rig that subjects one side to 34 ± 2 °C and >75% RH air and the other side to 77 K for either 9.5 or 69 h. The results demonstrated that the water content is mainly accumulated in the warm half for the 9.5 h cases, while it extends to about 80% of thickness for the 69 h cases. In consideration of the fact that the moisture uptake is mainly driven by cryopumping effects, which will be stronger as the temperature gradient is further enlarged. However, little information is now available on the intrusion of moisture into the foam material under liquid hydrogen (LH_2) temperatures.

This study dedicates to experimentally determine the moisture uptake of the PU foam specimens with the temperature on one side below 30 K. The PU foam was produced by Aerospace Research Institute of Materials and Processing Technology (ARIMT) of China. Two home-made single-stage pulse tube cryocoolers (PTC) in parallel with a cooling power of 15 W/20 K [15] each are used as the heat sink. On the other side, 303 ± 0.5 K and >95% relative humidity (RH) are maintained by using a positive temperature coefficient heater and the ultrasonic humidifier, respectively. A special fabrication was designed to minimize the heat leak to the PTCs from the ambient. The comparison of moisture uptake at 26 K and 79 K [13] is made and analyzed.

2. Materials and experimental apparatus

The tested materials and insulation structure are the same as the descriptions in our previous paper [13]. For completeness, a concise introduction is given here. The insulation structure consists of buffer layer, foam insulation layer and thermal protection layer from the tank wall outward. The buffer layer is actually a thin layer of epoxy cryogenic glue. The rigid foam layer is formed by a spray-on method with the machined thickness of 20 mm and density of about 48 kg/m^3. It has anisotropic closed-cell morphological structure with the cell diameter of about 60 μm. The outer surface of the foam layer is adhesively bonded by a thermal protection layer, which is a thin laminate of Kapton–aluminum–Kapton (KAK) and glass cloth.

Fig. 1 shows the schematic of the test apparatus, which mainly consists of three parts: the upper cryogenic system, the middle specimen fixing system and the below ambient temperature chamber (ATC). The cryogenic system mainly comprises two identical single-stage GM type PTCs, an oxygen-free copper stem, a corrugation plate and a vacuum chamber. Two cold ends (2) are thermally

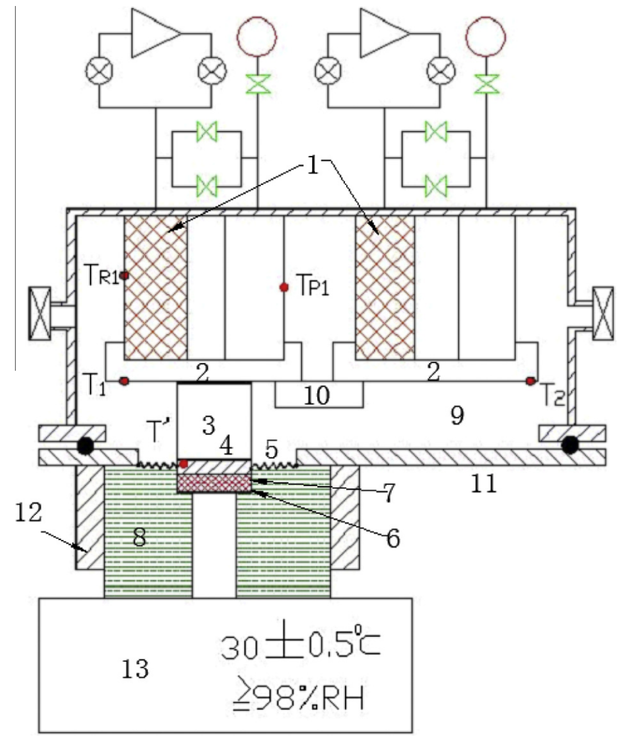

Fig. 1. Schematic of apparatus for moisture uptake of foam cooled by two pulse tube cryocoolers. (1) Pulse tube cryocooler; (2) Cold end; (3) Copper stem; (4) Copper disk; (5) Corrulation plate; (6) O-rings; (7) Specimen; (8) Support; (9) Vaccum; (10) Copper block; (11) Bottom plate; (12) LN_2 reservior; (13) Ambient temperaturn chamber (ATC).

bridged by a copper block (10). Indium films are filled between the copper block and each cold end for good thermal contact. The vacuum chamber consists of the above cuboidal cover and bottom plate (11), which are seal connected by a Teflon gasket instead of the O-ring, in consideration of the possible low temperature on the bottom plate. It is a challenge to transfer heat to the cryocoolers, which are installed in a vacuum chamber. A copper stem (3) with diameter of 80 mm and length of 40 mm is employed to connect the cold end (2) and a copper disk (4) with thickness of 8 mm and diameter of 80 mm. The contact surfaces of the copper stem are also covered by indium films in order to decrease the contact thermal resistance. Considering the thermal contraction during the cryogenic operation and also to decrease the conductive heat losses, a thin-wall corrugation plate (5) made of 304 stainless steel is specially designed and soldered with the copper disk (4) and the bottom plate. The corrugation plate can vertically move up and down slightly and withstand the pressure difference between the ambience and the vacuum.

The temperatures in each PTC cold end (T_1 and T_2) and middle of the regenerator (T_{R1}) and the pulse tube (T_{P1}), as well as the copper disk (T′) are measured by calibrated Rh–Fe resistance temperature sensors with accuracy of 0.1 K. Specifically, a short trough is made on the upper surface of the copper disk (4) to hold the sensor. The surface below the copper disk is relatively lower about 3 mm than the wave crest of the corrugation, to guarantee the specimen can be always pressed on the disk tightly.

It is necessary to minimize the heat leak through the fixing structure of the specimen in order to reach cryogenic temperatures. The heat leak is primarily due to: (a) the conduction in the vertical direction through the tested foam specimen (7); (b) the conduction in the radial direction through the corrugation plane (5) to the central copper disk (4); (c) the convective heat transfer

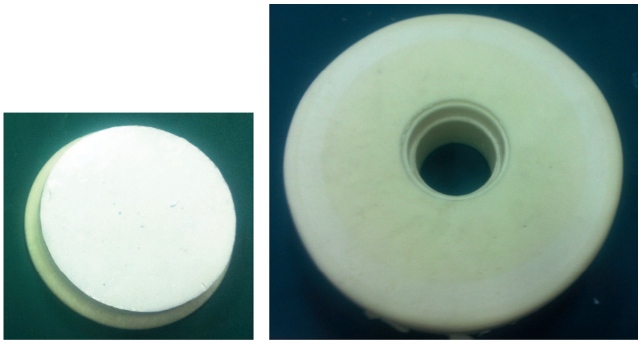

Fig. 2. Geometry and structure of specimen fitted onto the support.

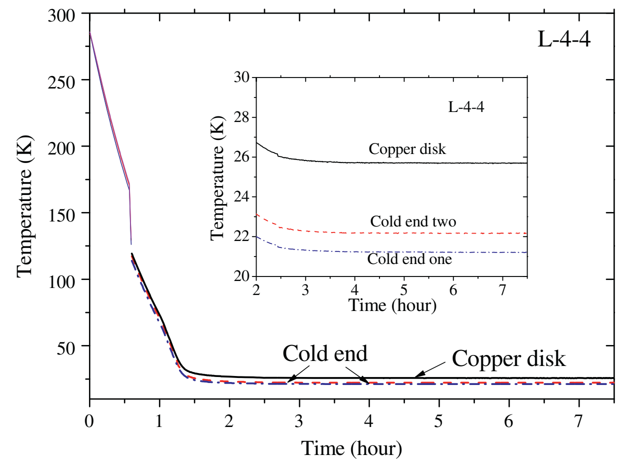

Fig. 3. Typical cool-down curves of copper disk and pulse tube cryocoolers.

of the liquid and gas air in the clearance between the specimen periphery and the inner surface of the support (8). Several key steps have been taken as follows: to minimize the heat leak due to (a), the effective diameter of the specimen is reduced to 70 mm from 200 mm for LN_2 temperature [13], while the nominal thickness of 20 mm remains unchanged. The specimen is machined to have a 4 mm thickness on a 10 mm wide periphery while maintaining an 80 mm diameter with the nominal 20 mm thickness, as shown in Fig. 2(a); to minimize the heat leak due to (b), the thermal conduction path should be lengthened; therefore, there are three corrugations in the 240 mm O.D plate with the corrugation pitch of 12 mm and the peak height of 6 mm; to minimize the heat leak due to (c), a large-diameter support with the same PU material as the specimen is made to support the specimen. It has an outer diameter of 280 mm and thickness of 100 mm. To tightly fix the specimen, from bottom up, there are three coaxial holes in sequence in the center of the support, one with diameter of 70 mm and height of 80 mm, one with diameter of 80 mm and height of 16 mm and one with diameter of 90 mm and height of 4 mm, as shown in Fig. 2(b). The large contact area between the upper face of the support and the corrugation plate greatly increases the flow resistance from the room-temperature environment to the cryogenic disk. An annular LN_2 reservoir is also installed in the support periphery to decrease the local air temperature. In addition, an O-ring is used between the below face of the specimen and the interior ladder of the support to stop air flow, and no KAK is used to cover the edge of the sample. The design is proved an effective way by the subsequent experiments that there is no ice formed at the periphery of the specimen. The design ensures that the moisture uptake is exactly incurred through the specimen surface exposed to the specific environment, rather than through the periphery.

Before a normal test, the support and the specimen is put on the top of the ATC, which can move and up vertically through the manual jack under it. The structure of ATC is same as our previous experiments under LN_2 temperature [13], except that the dimensions are reduced. And, the way to maintain the constant temperature and relative moisture in ATC are also same.

3. Test and weighing procedure

Three groups of samples are tested and they are IV-1, which is butt-jointed and no weathering period, IV-4, which is not butt-jointed and no weathering period, and L-4, which is not butt-jointed and has three months of weathering period under the direct sunlight in Beijing. The specimens are firstly stored at temperature of 303 K and relative humidity of 30% for over 48 h to guarantee a consistent starting point.

Particular care has to be paid to the experimental procedures to achieve reliable and reproducible data. When beginning a test, the specimen is firstly fixed into the support and tightly pressed onto the lower face of the copper disk by vertically moving up the ATC. Then one starts up the PTC system, including the vacuum pump, two independent compressors, and the measuring and controlling system. After about one and a half hour, the disk temperature will be cooled down to 30 K, which is considered as the start point of the moisture uptake experiments, and the temperature and humidity sustaining system in the ATC is triggered. Fig. 3 shows the typical cool-down curves during the experiments. There is a temperature difference of about 4 K between the copper disk and the main PTC cold end.

On reaching the specified test period, the PTCs are turned off. Different from the taking-off process for LN_2 temperature in our previous works [13], the specimen is firmly adhered to the copper disk, while the support can be easily taken off when moving down the ATC. The reason is mainly attributed to the formed solid oxygen and solid nitrogen in-between, which serve as the "adhesive". The adhered phenomenon of specimen by the solid air seems to be inevitable. Fortunately, when the temperature of copper disk ascends to about 65 K, slightly higher than the melting point of solid nitrogen (63.29 K, 10^5 Pa) in about 10 min, the specimen can be easily taken off without incurring any tiny difficulty.

As soon as the support is take off, the droplet of liquid air drops like rain, and the periphery of the specimen and the corrugation plate will be quickly covered by a layer of frost, as shown in

Fig. 4. Photo of specimen glued to the copper disk tightly when the supporter is just taken off.

Fig. 4. However, there is no ice found on the periphery of the specimen when the support is just taken off.

To avoid the added weight by the frost and the water droplet appended on the hot face, all the faces of the specimen are cleaned, and then, it is put on a laboratory balance (AL204, accuracy 0.1 mg, Mettler Toledo, Swiss) for weighing. Fig. 5 shows the typical curves of additional weight changes in the weighing process. Here, the value of additional weight due to moisture uptake is determined as follows:

$$\text{Additional weight } (\%) = (M_i - M_0)/M_0 \times 100, \quad (1)$$

where M_i is the temporal mass based on the entire weight of the specimen when it is weighed, and M_0 is the initial mass just before test.

Interestingly, the magnitude of additional weight during weighing does not keep constant and accelerates to decrease with time. The reasons can be qualitatively explained as follows: when the specimen is just put on the balance, the temperature on the cold face is still lower than the dew point of the ambient air, so the water vapor in the air will deposit until the temperature is high enough after heat transfer with the air. During this process, the weight increases. Simultaneously, on the hot face, the absorbed water just near the surface will re-vaporize by the convective mass transfer with the dry air nearby. During this process, the additional weight decreases. It is found that the effect of the later becomes predominant. However, it should be pointed out that the competition between the two processes is greatly affected by the interval between the moment the specimen left the rig and that being put on the balance. For our cases, the interval of less than 1 min in the room environment is guaranteed.

4. Results and analysis

For the calculations of additional weight, the maximum value in Fig. 5 are adopted as M_i and substituted into Eq. (1). Three specimens with different conditions are tested (see Table 1), and every case consists of at least two samples, each tested for both 5 h and 9 h. That is, the sample is firstly tested for 5 h, then after weighing, it is put into the temperature and humidity controlled equipment again for over 48 h to restore to the initial status. Followed, it is installed onto the rig for a 9-h test.

Fig. 6 shows the additional weight due to moisture uptake of IV-4 foam with 78 K or 26 K on one side and 30 °C, >95% RH on the other side. The data for 78 K cases are cited from our previous work [13]. The test result seems scattered and not accordant well with each other in the same case. The reason is primarily attributed to

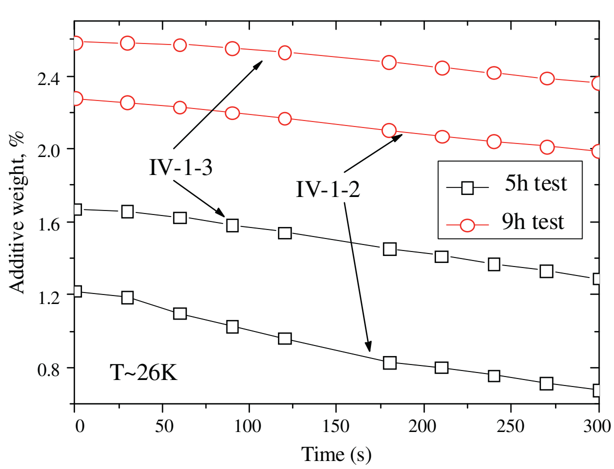

Fig. 5. Typical curves of additive weight vs. time during the weighing process of foam specimen.

the aforementioned uncontrollable weighing process. The performance of the manual wipe-off of the droplets and frost seriously depends on the judgment of the operators in the limited time. Thus, in consideration of the measuring errors, the scatter of the obtained data is considered to be acceptable. The maximum value of 9-h test for 26 K case is 5.63%, which is only a little larger than the value of 5.2% for the 78 K case. It is surprising that the moisture uptake of the PU foam is almost same under the test condition of 26 K and 78 K at cold side. The results will be further verified in the following part. The reasons can be qualitatively explained as follows: due to the closed cell structure of the foam, the mass diffusivity inside the interior is considered to be small during the test period. Therefore, the additional weight of the foam is primarily due to the moisture intrusion into the interior driven by the pressure difference as a result of cryopumping effect. When the temperature drops, the local pressure drops and the gases nearby are drawn to sustain the pressure balance. It had been considered that the larger is the temperature drop, the more moisture absorption will occur. The local temperature is actually dependent on the heat transfer performance. One hand, the effective thermal conductivity of PU foam with R141b as the blowing agent is quickly dropped from about 10 mW/m K at 78 K to less than 2 mW/m K at about 26 K, measured by Tseng et al. [6] under the conditions of constant pressure of 10^5 Pa. Although, in the temperature ranges in question, the effective conductivity will increase to about 12 mW/m K for the foam after moisture uptake [9], it is still relatively small. It implies that in the temperature range from 26 to 78 K, the temperature gradient is very large, or the thermal conductive distance is very short with constant heat flux. For example, the total heat flow is estimated to be 40 W from the performance curve of the PTC [15], and the conductive area is 3.85×10^{-3} m^2 (d = 70 mm), the averaged effective conductivity is estimated to be about 12 mW/m K, so the conductive distance from 26 to 78 K is calculated as only 0.06 mm according to the Fourier law. The results indicate that the solid oxygen and solid nitrogen will not form inside the foam, except in a very thin layer (∼0.06 mm) near the cold surface. On the other hand, Fesmire et al.'s [12] experiments of 8-h test at 78 K revealed that the water/ice/frost was found to exist mainly in one third of the outer warm side with progressively smaller amounts toward the cold side. It means that the moisture uptake of the foam mainly happens in the relative high-temperature zone near the hot side. The similar phenomenon is also observed by the 3D images of water content obtained by Vanderlaan et al. [14] using MRI. Therefore, the effect due to the further drop of temperature from 78 to 26 K at the cold side on the moisture uptake of the foam is limited.

Our previous works had proven that the crack in the foam will greatly deteriorate the final moisture uptake for the 78 K cases [13]. Here, the crack in the foam means that the whole cylindrical foam is split jointed by two identical half halves. The crack is filled with the epoxy glue, and then sealed by the thermal protection layer on the hot surface. The similar tests at 26 K situation are not performed; instead, the 20 mm- thick sample is butt-jointed by two same 10 mm-thick ones. Between the halves, the same KAK as being used in the hot surface is adhesively bonded. Fig. 7 compares the additional weight due to moisture uptake of the foam with and without the butt- joint when the cold side is at 26 K. Again, considering the measuring errors, the scatter of these two cases is accepted. And, the averaged value of the cases with the butt-joint is a littler smaller than the cases without the butt-joint, since the middle KAK helps to prevent the water vapor from penetrating through it. The results also indicate that the most of the water uptake occurs in the half of the foam to hot side in the limited period, as experimentally revealed by Fesmire et al. [12].

Fig. 8 compares the additional weight due to moisture uptake of PU foam with and without 3-month weathering period at 26 K

Table 1
Conditions and initial mass of the tested specimens.

Specimen	Conditions	Test time (h)	Temperature (K)	Initial mass (g)
IV-1-1	Butt-joint, no weathering time	5	25.3	5.8098
		9	25.9	5.7923
IV-1-2		5	25.7	6.2378
		9	25.6	6.1645
IV-1-3		5	25.7	6.4428
		9	25.9	6.4365
IV-4-1	No butt-joint, no weathering time	5	26.2	6.4319
		9	26.6	6.4207
IV-4-2		5	26.2	5.7607
		9	26.4	5.746
IV-4-3		5	26.3	4.6436
		9	26.6	4.6407
L-4-3	No butt- joint, 3 h of weathering time	5	25.9	5.235
		9	26.6	5.2393
L-4-4		5	25.7	5.1133
		9	25.9	5.1137

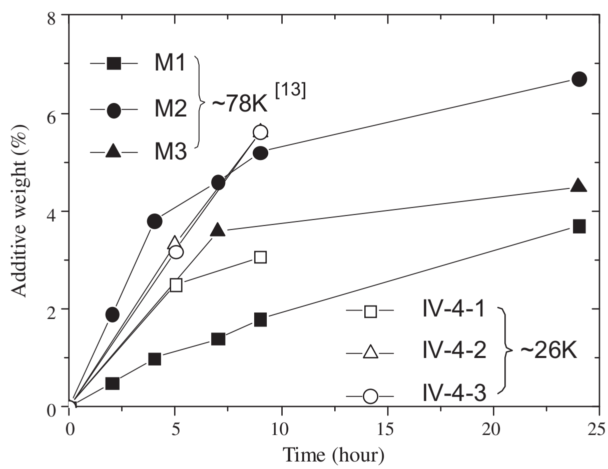

Fig. 6. Additive weight of PU foam without butt-joint and weathering time due to water uptake for 78 K or 26 K on one side and 30 °C, >95% RH on hot side.

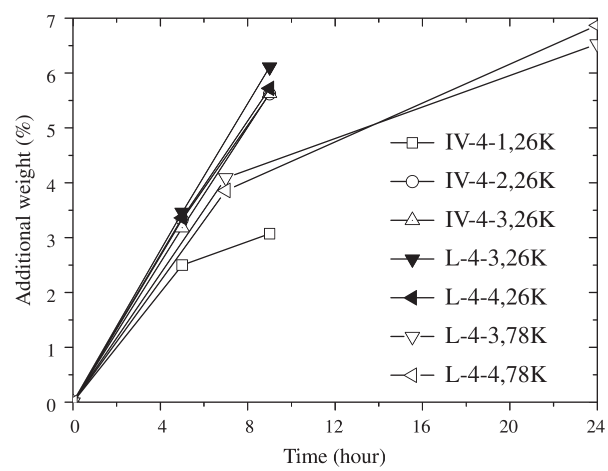

Fig. 8. Comparison of additional weight due to moisture uptake of PU foams with and without 3-month weathering time when 26 K on one side and 30 °C, >95% RH on hot side.

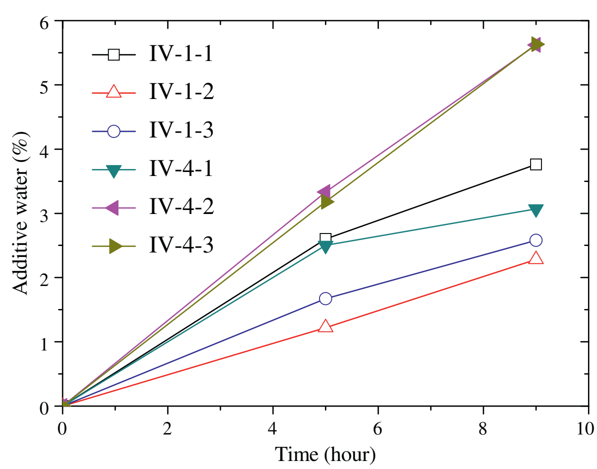

Fig. 7. Additive weight of PU foam with and without butt-joint due to water uptake for 26 K on one side and 30 °C, >95% RH on hot side.

conditions. It is found the additional weight of the foams with the weathering period increases more quickly than the one without the weathering period. The trends are consistent with the experimental results by Fesmire et al. [12] at 78 K conditions. It is explained by them that the foam material, although a closed-cell type, have some open-cell content and are not impermeable to water vapor, the weathering further degrades the internal structures. Compared with the results at 78 K by Fesmire et al. [12], our measuring values at 26 K is an order of magnitude smaller during the same test period because there is a thin layer of KAK in the hot side in our cases.

In addition, to further check the effects of temperature at one side on the moisture uptake, the L-4 case with 200 mm diameter are also tested for 7 h and 24 h on the 78 K apparatus, to which the detailed introduction can refer to [13], and the results are appended on Fig. 8. Again, the results give evidence that the weight gain due to moisture uptake is almost the same in the conditions of either 26 K or 78 K.

5. Conclusions

A novel experimental apparatus was designed and fabricated to measure the additional weight due to moisture uptake of the polyurethane foam. Two single-stage GM type pulse tube cryocoolers instead of liquid hydrogen were employed as a heat sink below 30 K. The specimens were exposed to the approximately 26 K on one side and 30 ± 0.5 °C, >95%RH on the other side within 5 h or 9 h. The tests for three groups of specimen, according to whether there were butt-joint and 3-month weathering period, were completed.

The effects of heat sink temperature on the moisture uptake are nearly identical for 78 K and 26 K cases. The reasons primarily attributed to the small thermal conductivity of the foam material at cryogenic temperature ranges. There is no significant difference on the additional weight due to moisture uptake in 9 h test time whether or not there is a butt-joint in middle of the foam. Most of the moisture uptake occurs in the warm half of the foam. Primarily due to the protection of the KAK in warm side of the foam, the foam experiencing 3-month weathering period will only incur a small increase in the additional weight due to moisture uptake, compared with the no weathering cases.

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