DEVELOPMENT OF REINFORCED POLYURETHANE FOAM INSULATOR USING HFCS AS BLOWING AGENT

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ABSTRACT

Polyurethane foams are widely used as insulation in many industrial fields, especially for the cryogenic fields that require good thermal insulating performance. Currently, Polyurethane foam reinforced by glassfiber (R-PUF) is used for the main insulator of the LNG cargo containment with the 1st barrier of corrugated membrane, and HCFCs have been used as blowing agents for the thermal performance of the R-PUF.

However, the use of HCFCs (Hydro Chloro Fluoro Carbons) has been banned gradually since they were known to deplete the Earth's ozone layer. In case of HCFC-141b, it has been restricted already in the leading countries since 2004 although its ODP (Ozone Depletion Potential) is 0.15, low value compared to that of CFC-11 (ODP = 1.0). Due to this trend, the research on the development of new blowing agents which can replace HCFCs is under way throughout the world. HFCs (Hydro Fluoro Carbons), HCs (Hydro Carbons), and CO_2 (Carbon dioxide) have been considered the qualified alternatives of HCFCs due to their zero ODP. [1-4]

This paper deals with the development of the environment-friendly insulator for the cargo containment of LNG ships. In this project we selected mixing of HFC-365mfc/HFC-227ea as the blowing agent considering the thermal performance of R-PUF.

R-PUFs blown by HFC-365mfc/HFC-227ea were produced using the continuous production lines of three insulation companies in South Korea. For the application of the R-PUF to LNG cargo containment, the mechanical properties, physical properties, thermal performances, thermal shock, and compressive strength change under the operating environment were evaluated. From the test results, it was shown that all the R-PUFs developed by 3 companies had the characteristics that can be used as the main insulator of LNG cargo containment. [5]

INTRODUCTION

R-PUF Insulator

Polyurethane foam (PUF) has a very wide application such as insulation materials, cushioning, automotive parts, and energy absorption materials. Especially for LNG fields, PUFs are used as a main insulation of LNG pipe and LNG storage tank and PUF reinforced by glassfiber (R-PUF) is used as the main insulation of LNG cargo containment.

Throughout the world much concern is concentrated on the environment friendly fuels due to the problems like ambient contamination caused by fossil fuels such as coal, oil etc. Because of this trend recently much more natural gas is consumed in many countries. Much amount of natural gas is exported from production place to consumption place in shape of liquid (LNG) via LNG carriers. Now many LNG carriers are in order. Since 1990s the order of LNG carriers with the cargo containment constituted of corrugated membrane and reinforced polyurethane foam insulator like GTT Mark Ⅲ has been increasing. [1]
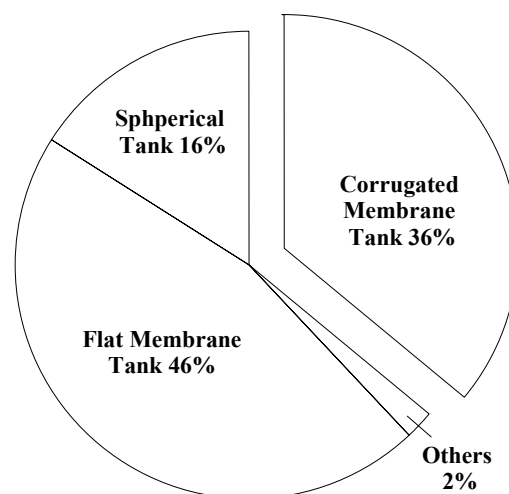
Figure 1. LNG Carriers on order - split by containment [1]

Largely there are two types of LNG cargo containment. One is self-supporting tank type like MOSS and the other is membrane tank type. Additionally membrane cargo containment can be classified into two systems. One is flat membrane tank type like GTT No 96 and the other is corrugated membrane tank type like GTT Mark Ⅲ. Flat membrane system is composed of the gas-tight inside barrier of invar and perlite box insulation. Corrugated membrane system is made up of the gas-tight inside barrier of STS 304 and R-PUF insulator. There is a large temperature difference over the insulator, about 180℃, between the inside LNG cargo containment and the ambient. In addition the insulator is subject to heavy loads caused by LNG static head, sloshing and the behavior of the ship, etc. It is very important to use qualified R-PUF insulator both to satisfy the boil off gas requirement and resist loads. [5]
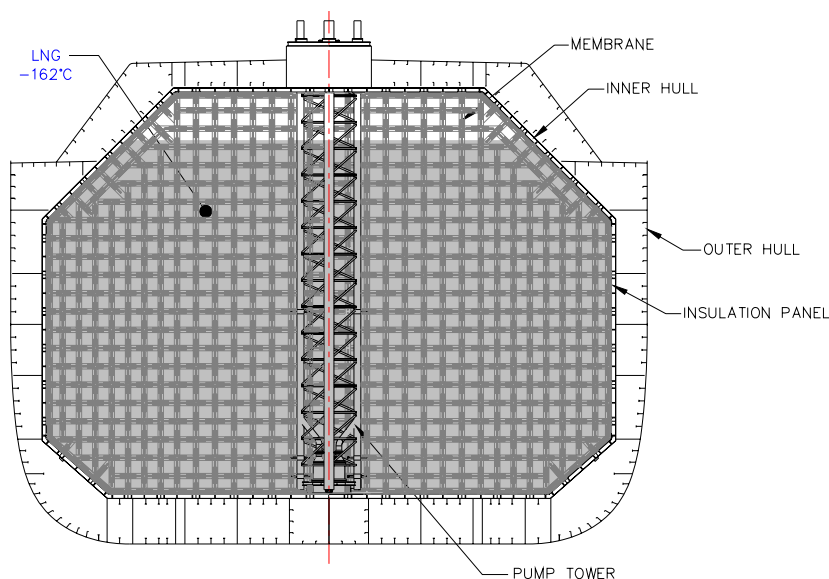
Figure 2. Schematic diagram of membrane type LNG cargo containment

Basic Formulations

The formation of polyurethane is based on the exothermic reaction between isocyanate and polyols. For the cell structure of polyurethane, physical and/or chemical blowing agents shall be used. Distilled water is one of the widely used chemical blowing agents. Distilled water reacts with isocyanate and generates carbon dioxide as shown in Figure 3. The widely used physical blowing agents are chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs). PUF formation is based on the reaction of isocyanate with polyol. The reaction is exothermic as shown in Figure 4. The reaction heat can be used to evaporate physical blowing agents and form a cellular structure.[2-4]
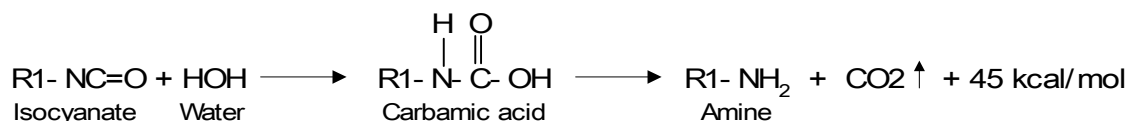

Figure 3. Reaction of isocyanate and water
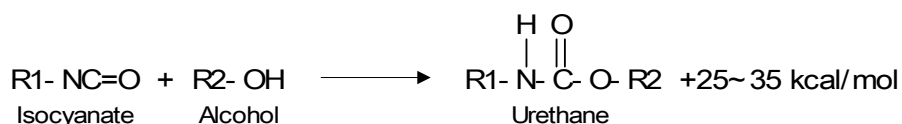
Figure 4. Formation of urethane

Blowing Agents

Fully halogenated chlorofluorocarbons (ex. CFC-11) have been used since the late 1950s as blowing agents for rigid polyurethane appliance foams. But since 1989 the production of these CFCs has been curtailed by legislation. In 1990s HCFCs, especially for HCFC-141b, had received highlight as a substitute for CFC-11 and had been used as blowing agents for PUF and R-PUF. HCFC-141b also has ozone depletion potential

(ODP=0.15) even though it is small compared to CFC-11 (ODP = 1.0). HCFC-141b has been restricted already in the leading countries since 2004. Limitations on the use of CFCs and HCFCs have arisen due to the environmental problems such as destruction of ozone layer and global warming.[4-5] Substitutes for CFCs and HCFCs such as hydrofluorocarbon (HFC), cyclopentane, and water have been under development. Their applications to cellular materials also have been studied.[6-9]

Table 1. Properties of various blowing agents [10]

Blowing gas	CFC-11	HCFC-141b	HFC-365mfc	HFC-245fa	Cyclopentane
Formula	CCl_3F	CCl_2FCH_3	$CF_3CH_2CF_2CH_3$	$CF_3CH_2CHF_2$	C_5H_{10}
Mol. Wt.	137.4	116.9	148.0	134.0	70.0
B.P. [°C]	23.7	32.1	40.0	15.3	49.3
Conductivity[1] [mW/m/K]	7.4	8.8	10.62	13.5	11.0
ODP	1.0	0.11	0	0	0
GWP	1.0	0.12	0.21	0.24	< 0.01

[1] Value of vapor at 10℃. 1998 UNEP Rigid and Flexible Foams Technical Options report, Table 1, unless otherwise noted.

[2] Aipfel et al, "HFC-365: A Versatile Blowing Agent for Rigid Polyurethane Foams", Polyurethanes Expo '98, 1998. Note, data at 25 °C

In this paper, Mixing of HFC-365mfc/HFC-227ea (93/7 volume ratio) was used as a blowing agent and R-PUF was produced in three insulation companies located in KOREA using commercial continuous line. The basic characteristics of R-PUF like compressive strength and thermal conductivity etc. were measured. Additionally the effects of operating environment to R-PUF were checked. As result, all the foamed R-PUFs showed enough thermal and mechanical capability to be used as main insulator for LNG cargo containment.

EXPERIMENTAL

Formulation

100 parts of polyol mixture of 3 kinds of polyether polyol and one kind of polyester polyol became base. Polymeric 4,4-diphenylmethane diisocyanate (PMDI) as isocyanate was used. In all systems, for complete reaction of polyol excess PMDI (NCO/OH = 1.20) was added. As blowing agent, about 0.5 parts of distilled water and 10 parts of mixture of HFC-365mfc/HFC-227ea were used. Additives such as 3 kinds of diamine catalysts, phosphoric flame retardant and polysiloxane ether surfactant etc. were added. 7 layers of glass fiber made by vetrotex was used as reinforcing agent.

Sample Preparations

Production. R-PUFs were produced using continuous production line. Largely there are three parts in the production of R-PUF. One is isocyanate part (liquid A) and another is polyol part (liquid B) with various additives like surfactant, catalyst, blowing agent etc. and the third is glass fiber part. In the production process, liquid A and liquid B is mixed evenly and the mixture is poured into 7 layers of glass fiber. As the reaction proceeds,

vaporization of blowing agents begins and the mixture swells with glass fiber. The key point of the reaction is accomplishing the even distribution of glass fiber. If distribution of glass fiber is biased, the deviation of mechanical property becomes large and the product can't be used as insulator of LNG cargo containment.

Before cutting test specimen, the produced R-PUFs were cured for at least 1 week at room temperature without exposure to sunlight. R-PUF are prepared by mixing some excess isocyanate and polyols with various additives such as catalysts, surfactant etc. General foaming process of R-PUF is shown in Figure 5. R-PUF used in measuring physical and mechanical properties are produced by commercial line with continuous dispending machine. R-PUFs were produced using high-pressure machines for good mixing between isocyanate and polyols. The amount of production is over 20 m long, with the width of 1.05 m and the foaming thickness of 230 ㎜. R-PUF boards were preliminarily cut by 3.0 m in length. R-PUF was produced two times in 2004 and 2005 and this paper is based on the second production in 2005.

Water	Catalyst	Polyols	Blowing agent	Additives

Blending → Conditioning / MDI → Mixing → Glass Fiber (7 Layers) ← Increasing Tensile Strength → Foaming → Curing → Measuring of properties

Figure 5. Production process of R-PUF

① outlet nozzle   ② glass fiber
③ bottom conveyor   ④ side conveyor
⑤ roller   ⑥ cutting facility
⑦ surface film   ⑧ bottom film

Figure 6. Continuous slab conveyor type production facility for R-PUF

(a)   (b)

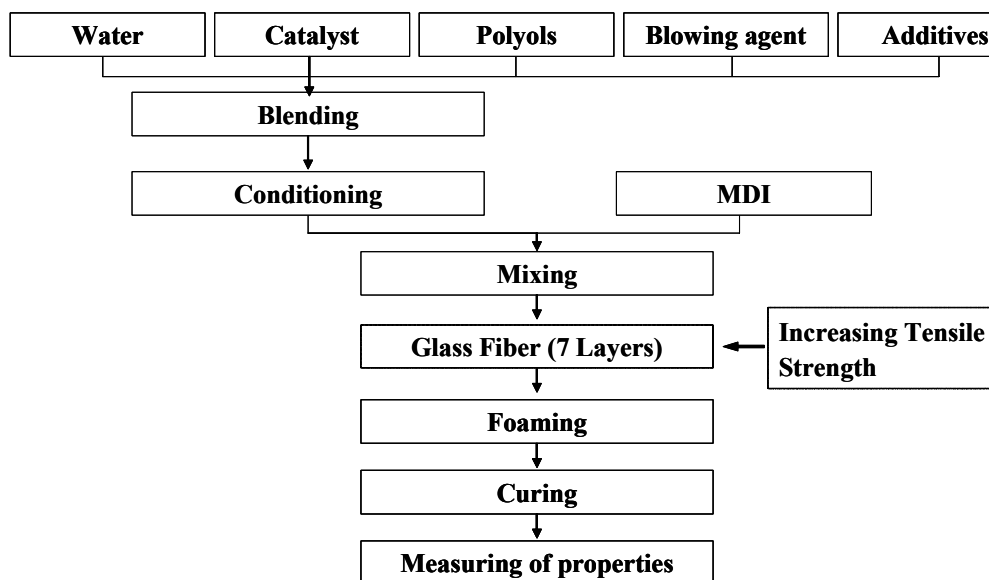**Figure 7. Sampling position; (a) physical and mechanical test, (b) evaluation of durable property**

Direction. Direction of R-PUF was defined as follows;

(a) x-direction: moving direction of outlet nozzle

(b) y-direction: production direction or moving direction of side/bottom conveyor

(c) z-direction: foam rising direction.

To check the distribution of glass fiber, test board were divided into three parts by height, top, middle and bottom. The same number of specimens except thermal shock, and strength change under the operating environment were taken from each part. Extent of glass fiber distribution was checked by deviation of physical and mechanical properties.
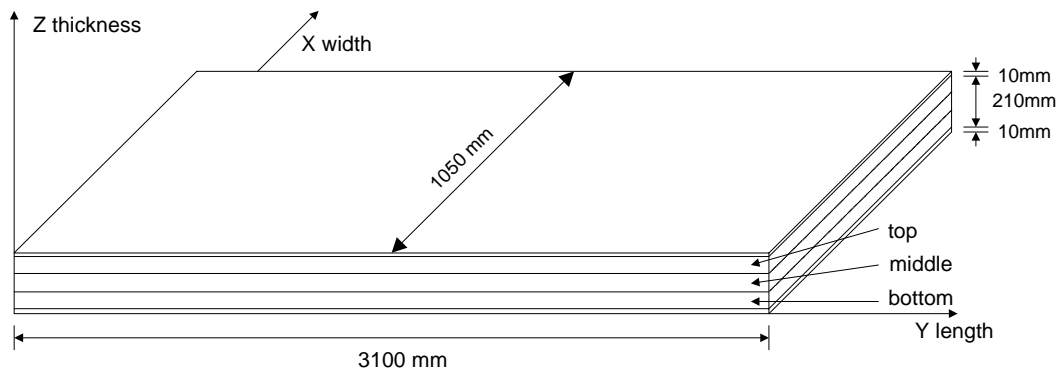
Figure 8. Direction of R-PUF board

RESULTS AND DISCUSSION

Physical Properties

Density, closed cell content, permeability, and CTE were measured. The test methods were summarized in Table 2 and the test results in Table 3.

Table 2. Summary of test method for physical properties

Item	Test method	Specimen size [㎜]	Instrument
Density	ASTM D1622	50 x 50 x 50	Vernier caliper
Closed cell content	ASTM D2856	25 x 25 x 25	Pycnometer
Permeability	ASTM E96	∅65 x 10	Humidity chamber
CTE	ASTM E228	5 x 5 x 50	Dilatometer

(a)   (b)

Figure 9. Instruments; (a) pycnometer, (b) dilatometer

Table 3. Test results of physical properties

Item			No. of specimen	A Company[1]	B Company[1]	C Company[1]
Density [kg/m³]		ave.	15	120	128	118
		dev.		2.7	2.0	2.1
		req.[2]		$117 < R^3 < 130$		
Closed cell content [vol%]		ave.	15	94.2	94.7	94.4
		dev.		0.88	0.41	0.57
		req.		R > 94		
Permeanbility [x $10^{-3} \mu$gm/Ns]		ave.	6	2.55	1.86	1.86
		dev.		0.27	0.19	0.27
		req.		R > 6.0		
CTE[4] [x 10^{-6} /℃]	x-dir.	ave.	15	22.4	21.5	22.2
		dev.		2.32	1.95	2.51
		req.		R > 35		
	y-dir.	ave.	15	23.3	22.3	22.7
		dev.		2.69	2.20	1.98
		req.		R > 35		
	z-dir.	ave.	15	58.4	55.3	57.9
		dev.		1.60	1.22	1.58
		req.		R > 65		

[1] Production of R-PUF was done in three companies in Korea, that is, Finetec, Kangrim Insu, Hankuk Carbon but abbreviation A, B, C doesn't indicate specific company.
[2] This value means the one needed for application to corrugated membrane type cargo tank.
[3] Average measured value
[4] Temperature range of CTE : -170℃ to 30℃

Mechanical Properties

Mechanical strength except tensile strength of R-PUFs was measured with an Instron (USA) 4467 universal testing machine (UTM) and a 3 ton load cell was used. Tensile strength was measured with a Kyungsung (KOREA) KSU-5M UTM and a 5 ton load cell was used. Tests were performed at ambient and cryogenic temperature in a temperature controlled chamber. To test mechanical strength at cryogenic temperature, all specimens except shear specimen were dipped into LN_2 chamber for at least 2 hours before testing. In case of shear test, specimens were put in the space where temperature is controlled around -100 ℃ using vaporization of LN_2. At testing, specimen was taken from the LN_2 chamber or the space and installed to jig in the chamber at -170 ℃ (-100 ℃ for shear test), and the door of chamber was closed. After 30 seconds (1 minutes for shear test), the test was started.

Compressive tests were performed according to ASTM D 1621. The size of the specimen was 50 mm (width) × 50 mm (length) × 50 mm (thickness), and the speed of crosshead movement was 5.00 mm/min. Tensile tests were performed according to ISO 1926 except sample size. The size of the specimens was shown in Figure 10. and the test speed was 5.00 mm/min. Shear tests were performed according to ASTM D732. The size of the specimen was 50 mm (width) × 50 mm (length) × 10 mm (thickness). The speed of crosshead movement was 1.25 mm/min. For each mechanical test, 10 specimens were tested and averaged. (c) Evaluation of durability; R-PUF's durability under operating environment was measured by affection of sea water, gas mixture of NH_3/N_2 (25/75 volume ratio) and LNG. For judgement, degradation of compressive strength was measured using the Instron USA 4467 UTM.
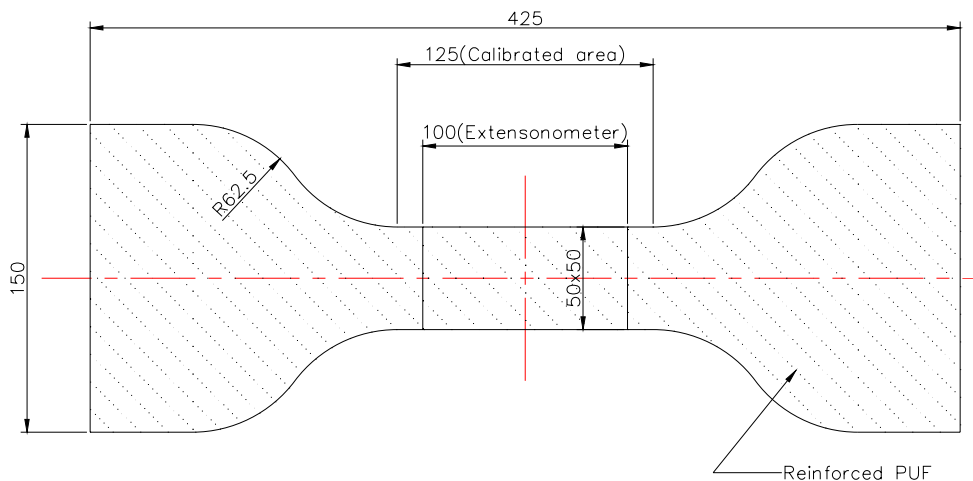
Figure 10. Specimen for tensile test

Table 4. Test results of compressive strength

Item		Direction[1]	No. of specimen	A Company	B Company	C Company
Compressive strength at 25°C [MPa]	ave.	z-dir.	15	1.28	1.44	1.31
	dev.			0.05	0.03	0.03
	req.			R ≥ 1.2		
Compressive strength at -170°C [MPa]	ave.	z-dir.	15	2.17	2.14	2.32
	dev.			0.09	0.11	0.15
	req.			R ≥ 2.0		

[1] Test direction is based on load direction to the specimen.

Table 5. Test results of tensile strength

Item		Direction	No. of specimen	A Company	B Company	C Company
Tensile Strength at 25°C [MPa]	ave.	x-dir.	15	3.24	3.00	3.38
	dev.			0.27	0.27	0.22
	req.			R ≥ 2.4		
Tensile Strength at 25°C [MPa]	ave.	y-dir.	15	3.26	2.97	3.09
	dev.			0.29	0.26	0.16
	req.			R ≥ 2.4		
Tensile Strength at -170°C [MPa]	ave.	x-dir.	15	3.95	3.36	3.90
	dev.			0.37	0.29	0.36
	req.			R ≥ 2.7		
Tensile Strength at -170°C [MPa]	ave.	y-dir.	15	3.96	3.43	3.78
	dev.			0.34	0.20	0.36
	req.			R ≥ 2.7		

Table 6. Test results of shear strength

Item		Direction	No. of specimen	A Company	B Company	C Company
Shear Strength at 25°C [MPa]	ave.	x-dir.	15	1.05	0.99	0.95
	dev.			0.08	0.06	0.03
	req.			R ≥ 0.9		
Shear Strength at 25°C [MPa]	ave.	y-dir.		1.03	0.99	1.00
	dev.			0.08	0.06	0.04
	req.			R ≥ 0.9		
Shear strength at 25°C [MPa]	ave.	z-dir.	15	1.78	1.98	1.91
	dev.			0.37	0.28	0.33
	req.			R ≥ 1.2		

Item		Direction	No. of specimen	A Company	B Company	C Company
Shear Strength at -100°C [MPa]	ave.	x-dir.		1.43	1.16	1.11
	dev.			0.14	0.10	0.09
	req.			R ≥ 1.0		
Shear Strength at -100°C [MPa]	ave.	y-dir.	15	1.46	1.21	1.29
	dev.			0.20	0.06	0.08
	req.			R ≥ 1.0		
Shear Strength at -100°C [MPa]	ave.	z-dir.		2.65	2.87	3.10
	dev.			0.29	0.32	0.30
	req.			R ≥ 1.4		

Insulation performance

Heat Transfer Through Foams. Thermal conductivity or λ-value is very important property in thermal insulations. Heat is transferred through foams via four mechanisms: ① gas conduction, Q_g, ② solid conduction, Q_s, ③ radiation, Q_r, and ④ convection, Q_c. The total heat flow, Qt, via the four parallel modes is normally assumed to be additive.

$$Q_t = Q_g + Q_s + Q_r + Q_c$$

The dominant mode of heat transfer in R-PUF is gas conduction until the temperature becomes low enough to liquefy the cellular gas. Solid conduction through R-PUF structure is small but not negligible for high density of R-PUF. Radiation is similarly a small component in R-PUF insulation for LNG industry. Convective heat transfer is negligible for foams with cell diameter under 3 ㎜. For an application of LNG industry, the effect of aging and gas permeability on λ-value must be considered, because R-PUF in cargo containment are used over 25 years. The total heat transfer through foam insulator is very dependent on the cellular gas content. The gas content is, in turn, dependent on age, permeability, and how the foam is shielded from the atmosphere. R-PUF would tend to lose a heavy gas, such as HFC-365mfc, very slowly but would absorb air several times rapidly. If unprotected, air will diffuse into R-PUF until the partial pressure of inside air is equal to outside. [12-15]

Aged Thermal Conductivity. For good evaluation of R-PUF blown by HFCs, both aged thermal conductivity and thermal conductivity curve shall be checked. The aged λ-values were measured with Netzsch Lambda 2000 series based on heat flow meter method. Thermal conductivity curve was measured with Netzsch GHP 456/1 based on guarded hot plate method.

The low λ-value of R-PUF is due to closed cell containing blowing gas such as HCFC-141b. R-PUF exposed to air shows an increase in λ-value with time. This is mainly because of the inward diffusion of air and the outward diffusion of blowing gas. The aged λ-value at 23℃ was measured according to ASTM C518, heat flow meter method with 40 ℃ temperature difference. To check the aging trend of thermal conductivity, the same specimen prepared during measuring the fresh thermal conductivity, was kept at ambient temperature without exposing to light. When the

specified time reached, thermal conductivity at 23℃ was measured using the specimen. Size of the specimen was 300 ㎜ (W) × 300 ㎜ (L) × 25 ㎜ (T). Two specimens were tested and average value is presented in Table 7.

Table 7. Aged thermal conductivity of R-PUFs

Item		A Company	B Company	C Company
λ-value at 23 °C [W/m/K]	Fresh	0.0272 (22)[1]	0.0286 (27)	0.0279 (25)
	2 months	0.0297 (62)	0.0303 (78)	0.0292 (107)
	4 months	0.0331 (122)	0.0322 (124)	0.0312 (155)
	6 months	0.0333 (220)	0.0332 (188)	0.0316 (216)

[1] Figure in the blank means the days elapsed after foaming.

λ-curve Between -160 ℃ and 20 ℃. Thermal conductivity curves between -160 ℃ and 20 ℃ are presented in Figure 11. Two specimens were used and the size was ∅ 200 ㎜ (diameter) × 25 ㎜ (thickness). Before testing, samples were installed to machine and conditioned by using vacuum. All the water and vapor were removed from the sample set. After conditioning the sample space was refilled with nitrogen gas. It took about 6 days to measure the λ-curve at six points from -160℃ to 30℃. The temperature difference throughout the measured temperature range was 40℃.
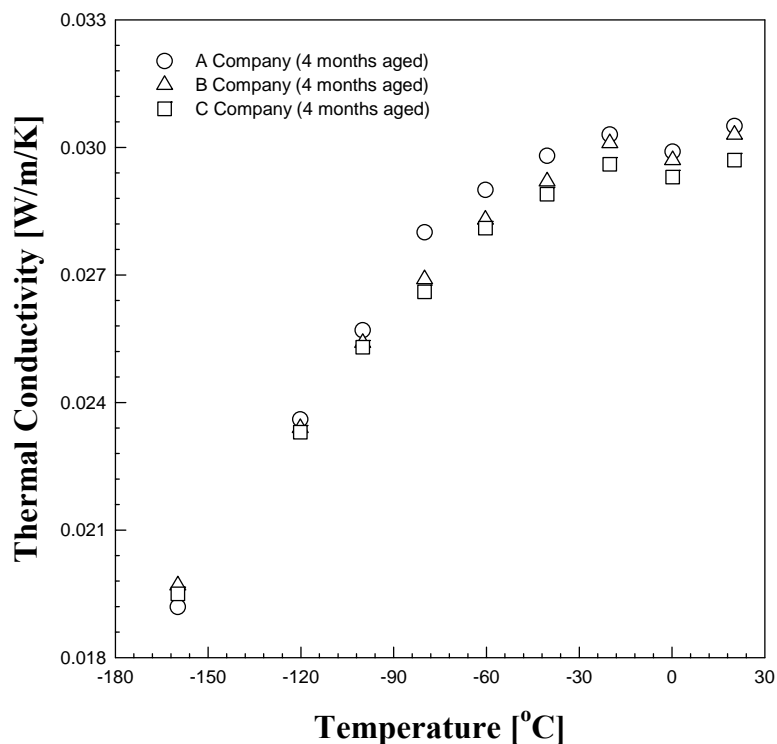
Figure 11. Thermal conductivity curve of R-PUF

Thermal Shock Test. Stability at cryogenic temperature was checked with a thermal shock test. The used specimen is shown in Figure 12. The thermal shock test consisted of three cycles. Each thermal shock cycle was like this; keeping the specimen inside of LN_2 vessel at -196 ℃ during one hour and heating the specimen by exposing to atmosphere during two hours. The first two cycles was followed by a reheating of the specimen. The check took place immediately after the third cycle as soon as the specimen was extracted from the LN_2 vessel. The specimen shall show no crack. Test results are shown in Table 8.

(a)   (b)
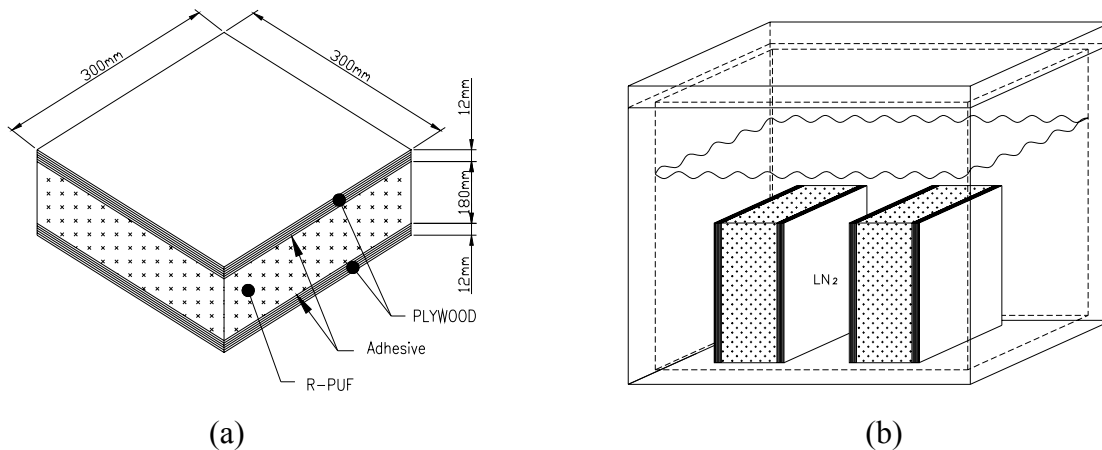
Figure 11. Thermal shock test; (a) specimen, (b) cryogenic chamber with LN_2

Table 8. Result of thermal shock test

Test item		A Company	B Company	C Company
Thermal shock test	1st cycle	○	○	○
	2nd cycle	○	○	○
	3rd cycle	○	○	○

where, '○' means no crack and '×' means crack.

Compressive Strength Change Under The Operating Environment. R-PUF is based on chemical materials like isocyanate, polyols. These chemical materials can experience change of mechanical strength under contact with seawater, sweeping gas, stored product etc. To evaluate the possibility, every 20 R-PUF samples were taken and average compressive strength of every 5 specimens was adopted as reference value. Remained 15 specimens were put under the various environments for long time. After the specified time elapsed, 2 weeks, 4 weeks, and 6 weeks, every 5 specimens were extracted from the tested space and was undergone testing of compressive strength. The ratio of compressive strength of 2 week, 4 week, 6 week to reference value was calculated and checked whether this value is not less than 0.8. The test results are shown in Table 9-11.
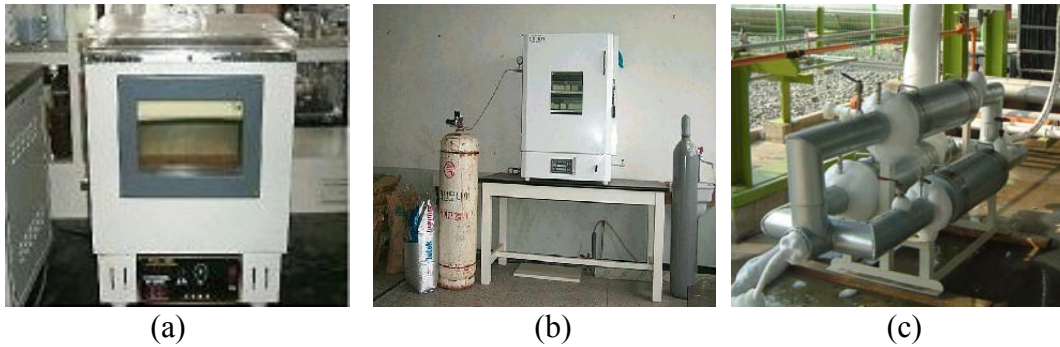
Figure 12. Instruments for strength; (a) sea water, (b) gas mixture, (c) LNG

Table 9. Test results of strength change under sea water

Item		No. of specimen	A Company	B Company	C Company
Compressive strength [reference]	ave.	5	1.25	1.38	1.34
	dev.		0.01	0.04	0.01
Compressive strength [2 weeks]	ave.	5	1.17	1.32	1.31
	dev.		0.03	0.03	0.01
	ratio		0.93	0.96	0.98
Compressive strength [4 weeks]	ave.	5	1.16	1.30	1.28
	dev.		0.03	0.06	0.02
	ratio		0.93	0.94	0.96
Compressive strength [6 weeks]	ave.	5	1.12	1.23	1.29
	dev.		0.03	0.06	0.03
	ratio		0.90	0.89	0.96

Table 10. Test results of strength change under gas mixture [NH_3/N_2=25/75 vol. ratio]

Item		No. of specimen	A Company	B Company	C Company
Compressive strength [reference]	ave.	5	1.25	1.38	1.34
	dev.		0.01	0.04	0.01
Compressive strength [2 weeks]	ave.	5	1.22	1.33	1.32
	dev.		0.06	0.07	0.01
	ratio		0.98	0.96	0.99
Compressive strength [4 weeks]	ave.	5	1.22	1.34	1.31
	dev.		0.06	0.03	0.03
	ratio		0.98	0.99	0.98
Compressive strength [6 weeks]	ave.	5	1.17	1.33	1.31
	dev.		0.04	0.02	0.05
	ratio		0.93	0.96	0.98

Table 11. Test results of strength change under LNG

Item		No. of specimen	A Company	B Company	C Company
Compressive strength [reference]	ave.	5	1.25	1.38	1.34
	dev.		0.01	0.04	0.01
Compressive strength [2 weeks]	ave.	5	1.21	1.36	1.30
	dev.		0.01	0.04	0.01
	ratio		0.96	0.99	0.97
Compressive strength [4 weeks]	ave.	5	1.21	1.35	1.29
	dev.		0.02	0.01	0.01
	ratio		0.96	0.98	0.96
Compressive strength [6 weeks]	ave.	5	1.19	1.33	1.29
	dev.		0.02	0.02	0.01
	ratio		0.95	0.96	0.96

CONCLUSIONS

All the physical properties of R-PUFs blown by HFCs, produced at three companies in South Korea were greater than the required values and showed small deviation.

The various mechanical strengths at room temperature and cryogenic temperature also were greater than the required ones and the deviations were not large. Deviations of the directions affected by glass fiber distribution were relatively larger than those of another direction but the difference was not large.

Thermal conductivity value at ambient temperature increased with the elapsed time after foaming until about 6 months but the increasing velocity decreased with time. After about 6 months thermal conductivity seemed to be stable and the changing amount was very small. From the curves of thermal conductivity all BOR values of three R-PUFs are lower than 0.15 %/day, the typical specification of LNG ship.

Decreases of compressive strength under various severe testing conditions were less than 20 %. From these test results strength change would not affect the safety of cargo containment.

From these test results, it is possible to use the R-PUF blown by HFCs as main insulator for LNG cargo containment of the corrugated membrane tank type.

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