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SUMMARY

A new concept for simplifying the application of radiation-shield insulation to cryogenic tanks was developed and tested for both ground and space performance. A vacuum-tight flexible casing of laminated aluminized Mylar encloses the multilayers, forming panels that are bonded to tank walls in an overlapping shingle arrangement. The panels, weighing only 0.34 pound per square foot (1.66 kg/m^2), are filled with carbon dioxide (CO_2) gas which cryopumps to provide required vacuum when the tank is filled with a cryogen. The thermal performance of this insulation was determined in flat-plate and cylindrical calorimeters. Of the many materials tested in the flat-plate calorimeter, polyurethane foam was chosen as the separator between radiation shields because of its small increase in heat flux when compressed to 14.7 psi (10.13 N/cm^2) in panels evacuated in the atmosphere. During ground tests, insulation panels as large as 3 by 6 feet (0.91 by 1.83 m) were mounted on a cylindrical calorimeter of liquid hydrogen and cryopumped to at least 5×10^{-3} torr ($6.66 \times 10^{-5} \text{ N/cm}^2$). This procedure resulted in a heat flux of 23 Btu per hour per square foot ($261\,000 \text{ J/(hr)(m}^2\text{)}$) for an external temperature of 70° F (294° K). In simulated space tests, panels evacuated by outside vacuum resulted in a heat flux of 0.86 Btu per hour per square foot ($9770 \text{ J/(hr)(m}^2\text{)}$) with outside surfaces at 70° F (294° K).

INTRODUCTION

This report describes the development and evaluation of a new concept to simplify the application of multiple radiation shields to cryogenic storage tanks.

Thermal protection on cryogenic propellant tanks of high-energy launch and space-propulsion vehicles is required to minimize fuel losses resulting from boiloff. Efficient

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insulation is particularly necessary for liquid hydrogen because of its extremely low boiling temperature and its relatively high ratio of wetted-tank surface to weight of liquid which result from its low density.

The development of a thermal protection system requires the solution of many problems. Since tank insulation imposes a payload weight penalty, lightweight insulating materials of sufficient strength must be provided. Structural problems also exist in providing the necessary attachment to the tank walls to withstand the launch environment. Although multiple thermal radiation shields separated by thin layers of low conductivity material are suitable for the vacuum environment of space, a more complicated insulation system is required during ground hold and boost through the atmosphere. In the atmosphere, air, which would degrade the insulation performance, must be prevented from cryopumping into the insulation either by purging the insulation with a noncondensable gas or by sealing the insulation with an air-tight barrier.

A number of multilayer insulation systems have been and are being investigated for hydrogen-fueled space-propulsion vehicles (refs. 1 to 4). The primary differences among the various investigations are the methods used to exclude air from the insulation during ground hold and the methods used to attach the multilayers to the propellant tank walls. Basic characteristics of multilayer insulation have also been investigated (ref. 5) to determine the fundamental properties of these insulations and to improve their thermal performance. Purging the multilayers with a noncondensable gas, such as helium (refs. 1 and 2), to exclude air results in high heat transfer through the insulation during ground hold because of the high thermal conductivity of the gas. The purged system also poses a venting problem during the external pressure decay from ground to space during launch. This problem prolongs the achievement of the required vacuum for long-term storage in space because of the slow escape of helium from within the insulation. By sealing the multilayers with an air-tight barrier, a vacuum can be established within the insulation, which lowers the heat transfer by gaseous conduction. Mechanically pumped static-type vacuum systems require impermeable vacuum casing materials to hold a vacuum for a long period of time. Or, if the systems are evacuated shortly before launch, bulky vacuum equipment is necessary at the launch site and on the flight vehicle.

A new multilayer insulation system that makes use of a cryopumping or self-evacuating principle when the insulation is exposed to a cryogenic environment was conceived and developed, in order to overcome the problems of mechanically evacuating the insulation. This insulation can be prefabricated in panel form and readily applied to cryogenic tanks. The development and evaluation of this insulation system, as presented herein, consisted of separate investigations of the major components. Included in these investigations were the basic multilayer insulation, the self-evacuation feature, the flexible vacuum casing material, and the conditioning of materials to reduce offgassing. These component results were then utilized in building large insulation panels, which

were installed on cryogenic tanks and evaluated for sealing capability, cryopumping, and thermal performance (ref. 6).

DESCRIPTION OF PANEL INSULATION SYSTEM

Basic Concept

A new insulation system concept, self-evaluated multilayer insulation (SEMI), utilizes panels of multiple-layer aluminized Mylar for radiation shields with each shield separated by a thin sheet of low conductivity polyurethane foam. Multilayer insulations consist basically of three elements: (1) multiple reflective shields, (2) low-thermal-conductivity shield separators between shield layers, and (3) low gas pressure (high vacuum) within the multilayers. Investigations have shown that these insulations can be highly effective in reducing the heat transfer

- (1) from radiation, by using highly reflective shield surfaces
- (2) from solid conduction, by using low-conductivity, low-surface-contact-area separators and by keeping compressive pressure on the multilayer stack as low as possible
- (3) from gaseous conduction, by using high vacuum within the multilayers.

The multilayers in this system are enclosed in a vacuum-tight flexible casing composed of a four-layer laminate of aluminized Mylar film (fig. 1). Before the panels are attached to the tank, they are evacuated to a very low air pressure and then backfilled with carbon dioxide (CO_2) gas to a very slight positive pressure (i. e., above atmospheric pressure). The prefabricated insulation panels are then attached to a tank in a

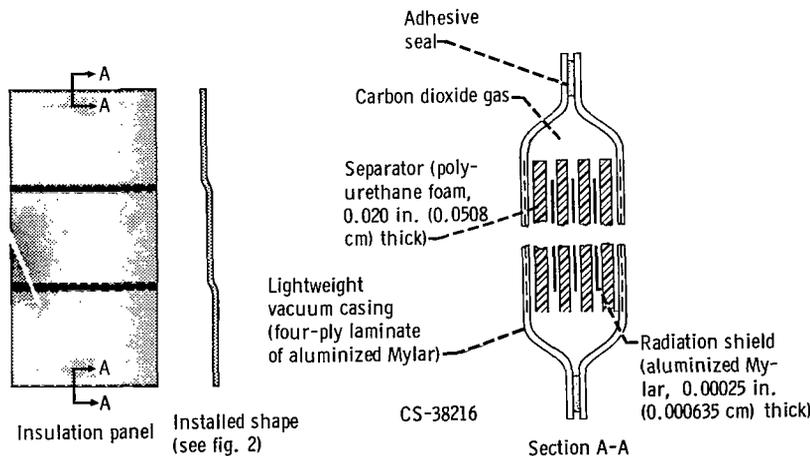


Figure 1. - Self-evacuated multilayer insulation (SEMI) panel for cryogenics.

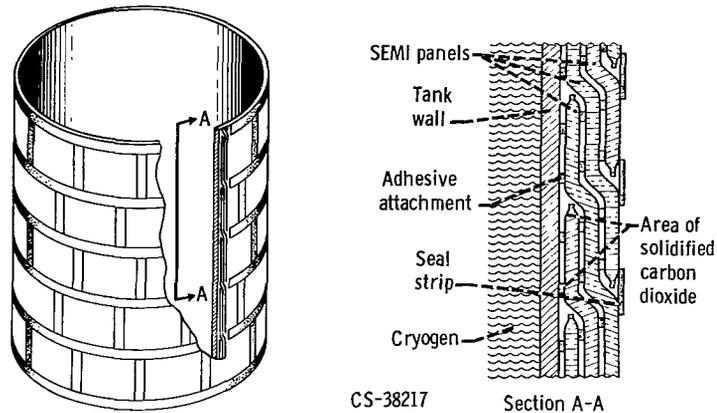


Figure 2. - SEMI panel installation using shingle arrangement on typical large propellant tank.

shingle arrangement with adjacent rows of overlapping panels (fig. 2). Each insulation-panel shingle can reasonably contain from 3 to 6 radiation shields, so that in the 3-panel shingle arrangement shown in figure 2, as many as 18 shields would form an effective thermal barrier. When the tank walls are cooled at the time of a cryogen filling operation, the CO_2 gas within the panels is solidified on the portion of the panel adjacent to the tank. Because of the low vapor pressure of solid CO_2 at cryogenic temperatures, the required vacuum within the insulation panels is achieved. During ground-hold operation then, the multilayers within the panel are subjected to a 1-atmosphere (10.13 N/cm^2) compression (approx. 15 psi).

Multilayer Insulation Materials

Radiation shields. - The basic requirements for the thermal radiation shields are light weight, low emissivity to ensure a low heat flux for the insulation system, the capability of withstanding cryogenic temperatures with little or no deterioration in emissivity, and low offgassing rates in a vacuum environment.

The thermal radiation shields for the insulation panels were made of 0.00025-inch (0.000635-cm) Mylar S film aluminized on both sides by a vacuum deposition process. The thickness of the aluminum deposition was such that the resulting electrical resistance was between 0.3 and 0.7 ohm per square on each side. Mylar film is light in weight (about one-half the weight of aluminum foil of the same thickness) and possesses relatively high strength and ductility, particularly at cryogenic temperatures. In addition, aluminizing produces a relatively low emissivity (from 0.014 to 0.077, ref. 5). The emissivity of the aluminized Mylar used in this system was from 0.022 to 0.032 at room temperature and from 0.018 to 0.022 at liquid-nitrogen temperature. No degradation of emissivity of the aluminized Mylar was noted after service in a panel.

Separators. - The basic requirements for a separator material between the radiation shields are lightweight, low thermal conductivity, minimum deflection under an atmospheric load, and the capability of withstanding repeated cycling of atmospheric loadings without breaking up or crumbling.

The separators placed between the thermal radiation shields of the insulation panels were 0.02-inch (0.0508-cm) thick sheets of rigid open-cell polyurethane foam with a density of about 2 pounds per cubic foot (32.04 kg/m^3). Open-cell foam was used to eliminate the possibility of trapped gases that may be present in closed-cell foam. Trapped gases within the foam could present a difficult problem at the time of panel evacuation because of a probable long duration of outgassing. These foam sheets were obtained by slicing large foam blocks with a horizontal band saw. The minimum thickness to which the foam blocks could be sliced and still allow the sheets to retain the necessary structural strength in bending and compression without crumbling was 0.02 inch (0.0508 cm).

Self-Evacuation System

Vacuum casing material. - The intended application of the panel insulation system dictates that the vacuum casing material used be light in weight, have extremely low permeability to air for providing vacuum integrity, remain flexible at cryogenic temperatures, and exhibit low edgewise thermal conductivity in order to minimize the lateral heat leak contribution through the shingled panel arrangement (fig. 2). Also required is an adhesive, for bonding the casing at the panel edges, that is compatible with the casing material and capable of flexing at cryogenic temperatures without developing cracks or leaks that would destroy the panel vacuum.

The casing material selected for the panel insulation system was a four-layer laminate of aluminized Mylar film. All four Mylar film sheets used in the laminate were 0.0005-inch (0.00127-cm) thick with the middle two layers aluminized on both sides and the outer two layers only aluminized on the inner side leaving the outer surfaces of the laminate nonaluminized. The casing laminates were bonded together at the panel edges with Narmco 7343/7139, a polyurethane-type adhesive, to completely enclose the multi-layer insulation.

Cryopumping gas. - The self-evacuation of insulation panels by cryopumping can be achieved with any gas which is condensable at the temperature present at the tank wall or the inner side of the panel. Carbon dioxide was selected for this system because in its pure state it has a vapor pressure of only 1×10^{-8} torr ($1.33 \times 10^{-10} \text{ N/cm}^2$) at a temperature of -320° F (77.3° K) (liquid-nitrogen temperature).

RESULTS AND DISCUSSION

Basic Multilayer Insulation

Compressive properties. - Room-temperature and liquid-nitrogen-temperature pressure-deflection tests were conducted on composite specimens for both open-cell and closed-cell polyurethane-foam separators with aluminized Mylar radiation shields. Each composite specimen, measuring 8.0 inches (20.32 cm) in diameter, consisted of four layers of foam sliced to a thickness of 0.020 ± 0.003 inch (0.0508 ± 0.0076 cm), with alternate layers of 0.00025-inch (0.000635-cm) aluminized Mylar.

The compressive strength was determined with the use of a pressure-deflection tester. Force on a compression plate that could be cooled with liquid nitrogen was provided by compressed gas operating on a piston. A dial indicator operated by the movement of the piston rod indicated the deflection of the test sample.

Figure 3 is a plot of the pressure-thickness characteristics at liquid-nitrogen temperature (-320° F (77.3° K)) for a representative specimen of both the open-cell and closed-cell foam. Both the first and second compression for each specimen are shown.

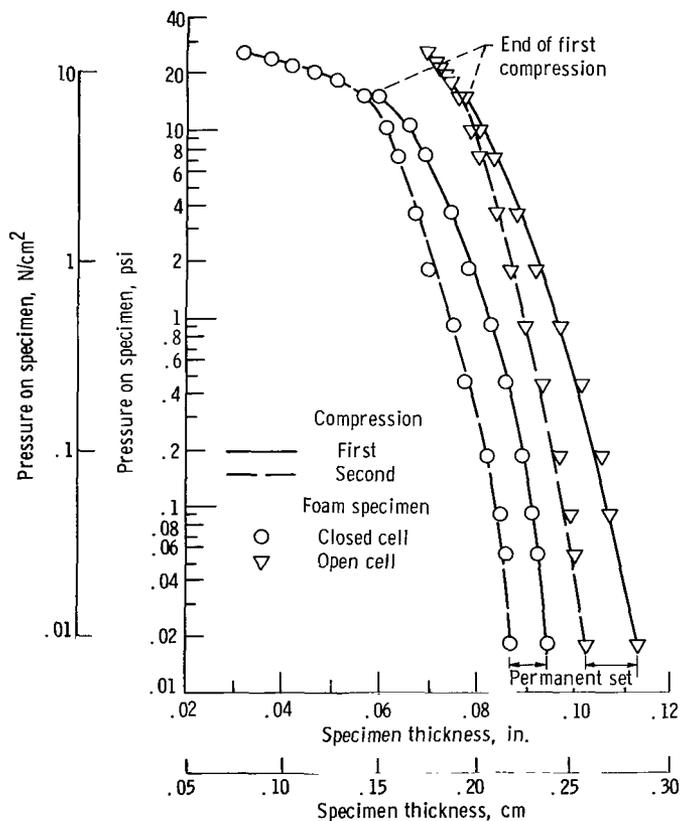


Figure 3. - Compressive properties of multilayer insulation specimens at -320° F (77.3° K).

The amount of permanent set was about 10 percent for either specimen after the first compression to 15 psi (10.34 N/cm²). For the second compression, the thickness at a pressure of 15 psi was about the same as that of the first compression.

Both the open-cell and closed-cell foam composite specimens exhibited more strength at liquid-nitrogen temperature than at room temperature. For example, the open-cell foam specimen at 15 psi (10.34 N/cm²) compressed to 0.062 inch (0.157 cm) at room temperature; at liquid-nitrogen temperature, it compressed to only 0.076 inch (0.193 cm). Similarly, the closed-cell foam specimen at 15 psi (10.34 N/cm²) compressed to 0.040 inch (0.102 cm) at room temperature and 0.059 inch (0.150 cm) at liquid-nitrogen temperature. The open-cell foam specimen exhibited greater strength and less deflection for compression to 26 psi (17.9 N/cm²).

Thermal tests with flat-plate calorimeters. - Thermal performance of the basic multilayer insulation was investigated with specimens in flat-plate calorimeters (ref. 7) using liquid hydrogen. The effects of compressive pressures from 0.001 to 1 atmosphere (0.01013 to 10.13 N/cm²) were studied on typical specimens (fig. 4).

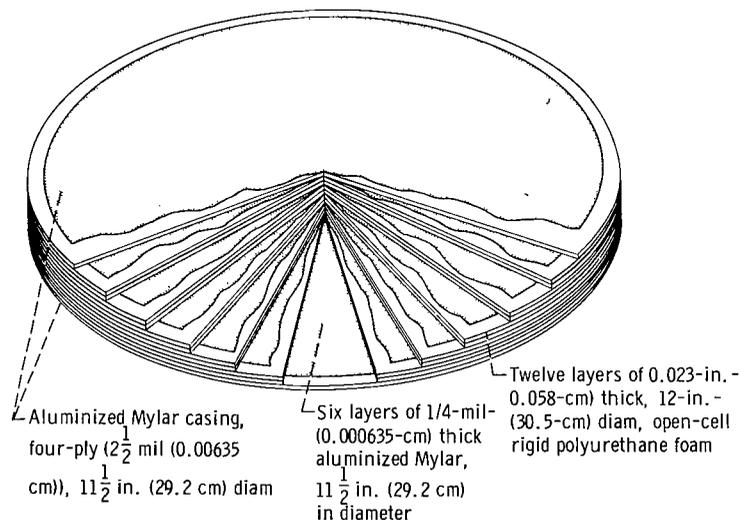


Figure 4. - Typical insulation specimen tested in flat-plate calorimeter.

The results of flat-plate calorimeter testing of several multilayer insulations with reflective shields and separators of different materials in a vacuum of 10⁻⁵ torr (1.33×10⁻⁷ N/cm²) is shown in figure 5. A foam separator between radiation shields exhibited the lowest increase in heat transfer from low compressive pressures to 15 psi (10.34 N/cm²). Interest in the effects of compressive pressure arises from the large deteriorations in insulation effectiveness that result from the application of only slight compressive pressures. Heat flux for some materials was as much as 100 times greater at 1 psi (0.689 N/cm²) than at the uncompressed condition. Compressive pressure on the insulation system during the ground-hold condition occurs as the result of atmospher-

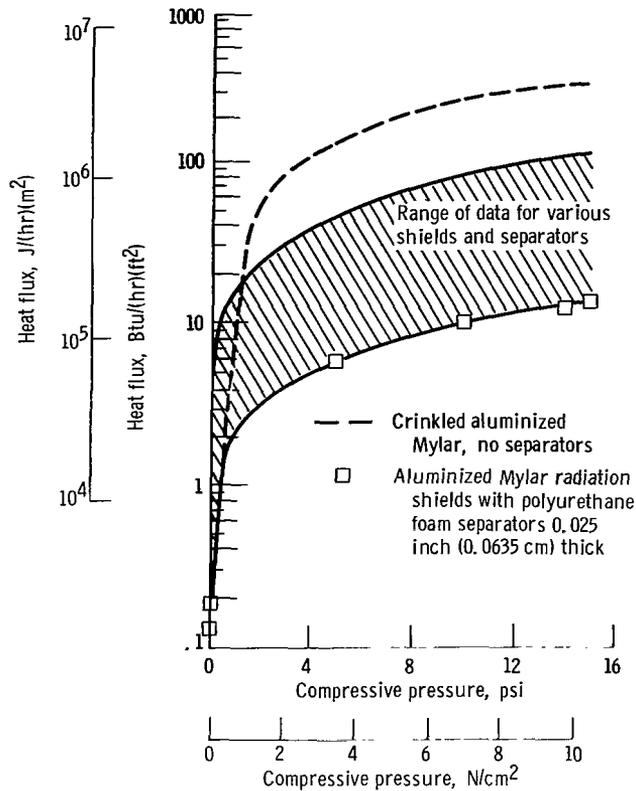


Figure 5. - Flat-plate calorimeter data showing effect of compressive pressure on heat flux through several multilayer insulations having reflective shields and separators of different materials in vacuum of 10^{-5} torr (1.33×10^{-7} N/cm²).

ic pressure exerted on the evacuated panels. Compressive pressure on the insulation system during space conditions results entirely from forces imposed by the casing system because of panel installation techniques. The main reason for choosing foam as the separator for the panel system was the fact that it exhibited the best thermal performance under the full range of compressive pressures.

An insulation specimen (fig. 4) was tested that would be exactly the same as the final three-panel insulation system; that is, each of the three panels contained 2 sheets of the four-ply aluminized Mylar casing material, 6 aluminized Mylar radiation shields, and 12 open-cell polyurethane foam separators. An additional separator was used between the shields to provide low heat transfer at ground-hold conditions. Three successive compressive pressure cycles indicated some degradation at low compressive pressures for each additional cycle. For the third cycle, at a compressive pressure of 0.01 psi (0.007 N/cm²), which simulates an estimated space condition for installed panels, the heat flux measured 0.33 Btu per hour per square foot (3740 J/(hr)(m²)) with a warm-side temperature of 70° F (294° K). The heat flux for the ground-hold compress-

sed condition at 1 atmosphere (10.13 N/cm^2) remained nearly the same for all three cycles.

Heat transfer as a function of vacuum changes within an uncompressed multilayer insulation specimen was also investigated in the flat-plate calorimeter by using a controlled pressure of helium gas in the sample chamber. This study was made to determine the vacuum level necessary in the self-evacuated panels to nearly eliminate heat transfer by gaseous conduction. Data shown in figure 6 indicate that a vacuum of 10^{-4}

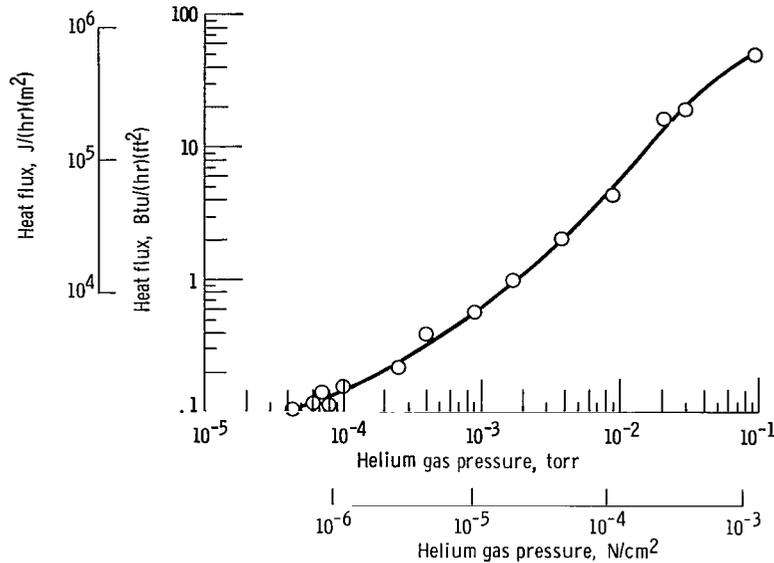


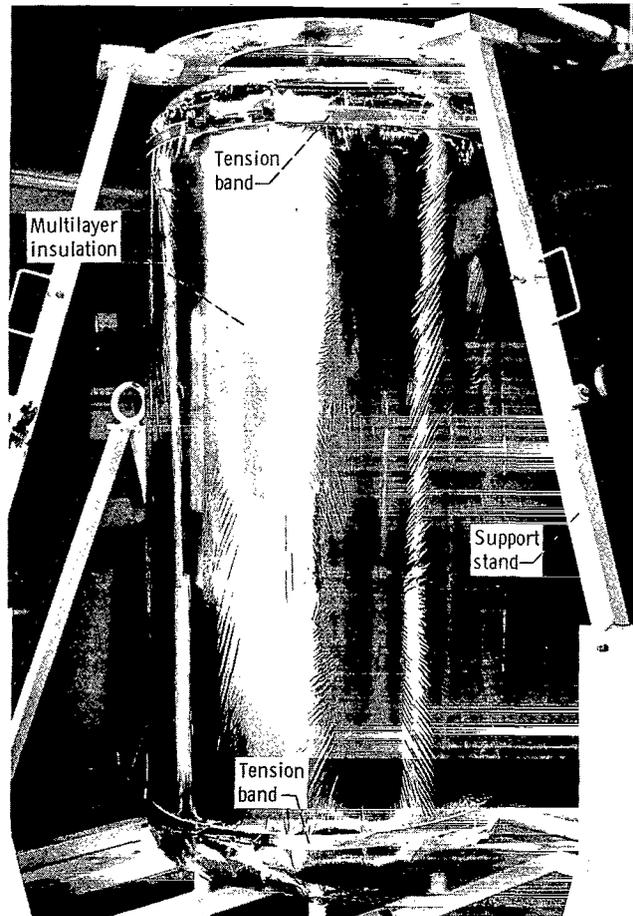
Figure 6. - Flat-plate calorimeter data showing effect of helium gas pressure on heat flux through multilayer insulation with reflective shields of aluminized Mylar and foam separators under no compressive pressure. Liquid hydrogen in calorimeter; warm-side temperature, 70° F (294° K).

torr ($1.33 \times 10^{-6} \text{ N/cm}^2$) should be present in the panels to achieve a heat flux lower than that resulting from a small compressive pressure condition at vacuums in the 10^{-5} torr ($1.33 \times 10^{-7} \text{ N/cm}^2$) range ($<0.33 \text{ Btu/(hr)(sq ft)}$ or $<3740 \text{ J/(hr)(m}^2\text{)}$).

Thermal tests with cylindrical calorimeter. - The basic multilayer insulation was evaluated on a cylindrical calorimeter to compare the heat flux under space conditions with that measured on the flat-plate calorimeter. Another objective was to obtain a heat flux for a nearly ideal multilayer installation that could later be compared with that measured for the self-evacuated insulation-panel system. The calorimeter tank was 30 inches (76.2 cm) in diameter and approximately 5 feet (1.52 m) in overall length including two guard chambers which were located at either end of a 3-foot- (0.914-m) long test section. The multilayer insulation applied to this tank consisted of 10 layers each of 0.020-inch- (0.0508-cm) thick open-cell polyurethane foam spacer and 0.00025-inch (0.000635-cm) Mylar radiation shields aluminized on both sides. No vacuum or

purge capabilities were required for the system because the tests were conducted in a large vacuum chamber. The insulation was applied to the cylindrical tank by using a layer of aluminized Mylar and a layer of foam as a composite sheet in a continuous circumferential wrap. The head insulation was interweaved with the cylindrical insulation so that intimate contact was achieved between the respective layers of Mylar and foam. No specific layer density was specified. Attempts were made, however, to install the insulation with minimum wrinkling, yet without visibly compressing the insulation. The completely insulated tank with tension bands installed at top and bottom to hold the insulation in place is shown in figure 7.

The insulated calorimeter tank was installed in a space chamber facility at the NASA Plum Brook Station. Thermal tests with liquid hydrogen in both the test tank and guard chambers were conducted for 12 hours. The guard tanks were maintained at a slightly higher pressure than the test tank to preclude the possibility that the guard tanks would recondense the cold vapors from the test tank used to calculate heat flux.



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Figure 7. - Basic multilayer insulation installed on calorimeter tank.

By using the data obtained during the latter part of the test run, the measured tank flow rate, corrected to 1 atmosphere (10.13 N/cm^2) absolute pressure and 70° F (294° K), was 8.52 cubic feet (0.241 m^3) per hour. A value of 1.003 Btu per cubic foot (37500 J/m^3) of hydrogen gas for the heat of vaporization of liquid hydrogen and a surface area of 23.6 square feet (2.19 m^2) were used to calculate the heat flux of 0.36 Btu per hour per square foot ($4090 \text{ J/(hr)(m}^2\text{)})$ for this 10-layer configuration. The thermal conductivity, based on this heat flux, a measured thickness of 0.54 inch (1.37 cm), and a surface temperature of 72° F (296° K) is 0.395×10^{-3} Btu per inch per hour per square foot per $^\circ \text{ F}$ ($20.5 \text{ (J)(cm)/(hr)(m}^2\text{)(}^\circ \text{K)}$). This heat flux corresponds closely to that measured in the flat-plate calorimeters for the multilayer insulation under a slight compressive pressure of 0.01 psi (0.00689 N/cm^2), as discussed previously.

Vacuum Casing Materials

Permeability and thermal conductivity of candidate materials. - Selection of a thin film or laminate for use as a vacuum casing material requires that properties of various material candidates be determined. Single-layer films, although excellent from an edgewise (parallel) thermal conductivity standpoint, were determined to be unacceptable from a permeability standpoint. Table I presents a number of laminated casing material candidates with their respective measured permeability. Also included in table I, is the product of thermal conductivity parallel to the layers times the material thickness, which allows comparison of materials on the basis of thermal performance.

Three classes of material appeared to be satisfactory for use as vacuum casing materials, namely, laminates of aluminized Mylar, a Mylar-lead-Mylar laminate, and

TABLE I. - PERMEABILITY AND LATERAL THERMAL CONDUCTIVITY PARAMETERS FOR POSSIBLE VACUUM CASING MATERIALS

Casing material ^a	Material thickness		Helium permeability (at STP)		Thermal conductivity times thickness	
	in.	cm	$\text{cm}^3/(\text{sec})(\text{ft}^2)(\text{atm})$	$\text{cm}^3/(\text{sec})(\text{m}^2)(\text{N/cm}^2)$	$\text{Btu}/(\text{hr})(^\circ \text{F})$	$\text{J}/(\text{hr})(^\circ \text{K})$
Mylar laminate (M/M)	0.002	0.00508	$>2.00 \times 10^{-4}$	$>2.12 \times 10^{-4}$	1.46×10^{-7}	2.77×10^{-4}
Aluminized Mylar laminate (Ma/aM/aM)	.0015	.00381	2.00×10^{-5}	2.12×10^{-5}	8.04×10^{-5}	1.52×10^{-1}
Mylar-lead-Mylar laminate (M/L/M)	.0028	.00711	$<2.18 \times 10^{-9}$	$<2.32 \times 10^{-9}$	1.33×10^{-3}	2.52×10^0
Mylar-aluminum-Mylar laminate (M/A/M)	.00135	.00343	$<2.18 \times 10^{-9}$	$<2.32 \times 10^{-9}$	3.75×10^{-3}	7.11×10^0
M/A/A/M	.0017	.00432	$<2.18 \times 10^{-9}$	$<2.32 \times 10^{-9}$	7.50×10^{-3}	1.42×10^1
M/A/M/A/M	.0032	.00812	$<2.18 \times 10^{-9}$	$<2.32 \times 10^{-9}$	7.50×10^{-3}	1.42×10^1
Ma/aMa/aMa/aM	.0025	.00635	2.98×10^{-6}	3.16×10^{-6}	3.22×10^{-4}	6.11×10^{-1}
Ma/aMa/aMa/aM/Ma/aMa/aMa/aM	.005	.01270	1.36×10^{-6}	1.44×10^{-6}	6.44×10^{-4}	1.22×10^0

^aM, Mylar film; A, aluminum foil; L, lead foil; a, aluminized surface; (/) denotes adhesive bond line between layers.

laminates of Mylar and aluminum. Based on a compromise of the thermal analysis and the requirements of low weight and sufficiently low permeability for achieving an acceptable vacuum performance, it was decided to employ the four-ply aluminized Mylar laminate casing material for the test insulation panels.

Adhesive evaluation. - Tests were conducted to determine the most suitable adhesive for sealing the casing material joints, which must be flexed when cooled to cryogenic temperature while vacuum tightness is maintained. Because all the candidate casing materials have a plain Mylar outside surface, only Mylar-to-Mylar joints were evaluated. Adhesive tests were conducted on sample small-scale insulation panels fabricated from two 13-inch- (33.0-cm) square pieces of Mylar-lead-Mylar laminate casing material with an evacuation tube attached to the top casing. Dexiglas was used between the casing material to provide a separation so that the panel could be easily evacuated. A 1-inch- (2.54-cm) wide adhesive joint was made on all the panels, and after adhesive cure, this joint was trimmed to a width of 1/2 inch (1.27 cm), making the sample panel 12 inches (30.5 cm) square.

Of the eight adhesives tested, the Narmco 7343/7139 appeared to be the most satisfactory and was used on the full-size calorimeter-tank insulation panels. This adhesive exhibited a low initial leak rate, and, after flexing in liquid nitrogen, exhibited little increase in leak rate.

Cryopumping of Carbon Dioxide Gas

Impurities study. - The maximum amount of noncondensable gas that can be initially present in the CO₂ was calculated to be only 0.132 parts per million in order to achieve a desired pressure of 1×10^{-4} torr (1.33×10^{-6} N/cm²) after cryopumping if no cryotrapping occurs. With cryotrapping, a phenomenon in which noncondensable gases are physically trapped or absorbed in a solidified gas, the percentage of noncondensables may be somewhat greater, and still the system can achieve a pressure of 1×10^{-4} torr (1.33×10^{-6} N/cm²). However, it is difficult to estimate how much noncondensable gas will be cryotrapped.

Coleman Instrument Grade CO₂ was used in this investigation. This grade has a stated purity by volume of 99.99 percent, which is less than 100 parts per million of noncondensables. Analysis of the actual cylinder of Coleman Grade CO₂ yielded the following results: hydrogen, helium, and neon were not detected (scale sensitivity of 0.2 ppm); oxygen and argon were not detected (scale sensitivity of 0.5 ppm); nitrogen was detected and had a concentration of approximately 25 parts per million. With this nitrogen concentration, the calculated cryopumped pressure at liquid-nitrogen temperature would be 2×10^{-2} torr (2.66×10^{-4} N/cm²), if none of the nitrogen was cryotrapped and if there is

assumed to be no change in pressure with temperature. Experimentally, however, it was determined in a small Dewar (a cryogenic container surrounded by an outer jacket) that Coleman grade CO₂ between the walls of the container and the jacket would cryopump from 760 torr (1 atm (10.13 N/cm²)) to 2.5×10⁻⁵ torr (3.33×10⁻⁷ N/cm²) in a 40-minute time period with either liquid nitrogen or liquid hydrogen. This result indicated that cryotrapping occurred. This acceptable low pressure showed that Coleman grade CO₂ was a suitable gas for this insulation system.

Although this low cryopumped pressure was finally achieved, initial attempts to cryopump CO₂ were not successful. It was determined in subsequent testing that the electronic gaging, namely, a cold cathode vacuum gage and a thermocouple-type vacuum gage, should not be operated at high CO₂ pressure because CO₂ gas decomposes into carbon monoxide and oxygen. At liquid-hydrogen temperature, carbon monoxide has a vapor pressure greater than 0.5 atmospheres (5.06 N/cm²). Low cryopumped pressures were achieved, however, when the gages were not activated until after a period of time to assure that the pressure had dropped below 1 torr (0.0133 N/cm²).

Substrate material investigation. - Tests to evaluate various materials for use as a CO₂ cryopumping substrate were conducted. A cryopumping substrate is the material on or in which the CO₂ solidifies on the cold side of the insulation panel. Tests with the small Dewar (employed in the impurities study) were conducted on sliced open-cell foam (spacer material) and on a woven fiberglass cloth (style 112, 3-mil (0.00762-cm) thick, coarse weave, 40 by 39 ends/in. or 15.7 by 15.4 ends/cm). These materials, in separate tests, were attached to the cold walls of the Dewar. In addition, a test using the bare walls to cryopump CO₂ was conducted for comparison. These tests were conducted by using liquid nitrogen in the inner container and Coleman grade CO₂ in the space between walls. The Dewar was mounted on a shake table to provide and maintain a vibration level of 6g (peak to peak) at 40 cycles per second throughout the testing.

Cryopumping tests indicated similar pumpdown times for both substrate materials with bare chamber walls and pressures to 1×10⁻⁴ torr (1.33×10⁻⁶ N/cm²). No problems were apparent in cryopumping to a pressure of 2×10⁻⁵ torr (2.66×10⁻⁷ N/cm²). Pressure spikes were consistently below 1×10⁻⁴ torr (1.33×10⁻⁶ N/cm²) for all tests when the specimens were vibrated. Therefore, the degree of spalling of the solid CO₂ and subsequent revaporization resulting in a pressure rise did not appear to be a problem.

From these tests, it was concluded that the open-cell foam spacer itself could be used as a substrate surface for cryopumped CO₂, and thus an additional material in the system is not required.

Gas flow conductance. - Carbon dioxide gas conductance measurements for both the longitudinal and normal flow directions in the insulation panels were necessary to assure that the self-evacuation rate of the panels would be acceptable. Insulation panels were fabricated of three Mylar radiation shields and four alternate layers of foam separators

0.02 inch (0.0508 cm) thick. The relative capability for longitudinal gas flow conductance was determined for these panels with cryopumping substrates consisting of (1) a single layer of foam on one side, (2) a two-layer thickness of foam added to one side, and (3) a 0.016-inch- (0.0406-cm) thick woven-glass layer added to the single layer of foam on one side.

Only minor variations were noted in the conductance of the three panels in the transition and viscous flow regimes, with experimental error probably accounting for the limited scatter encountered in the molecular flow regime data. The measured conductance for average panel pressures below 1 torr (0.0133 N/cm^2) was typically in the range 5×10^{-3} cubic feet per minute per foot of length per foot of width ($1.52 \times 10^{-3} \text{ cu m}/(\text{min})(\text{m length})(\text{m width})$) for these 0.08-inch- (0.203-cm) thick panels.

Gas flow around the inside edge of the insulation panels (normal flow direction) was also investigated. Insulation panels 2 feet long by 3 inches wide (70.0 by 7.62 cm) were fabricated by using straight or notched edges of the radiation shields that were flush with the edges of the foam separators. Panels with notched radiation-shield edges exhibited conductance greater by approximately a factor of 3 than panels with straight radiation-shield edges. The measured edge conductance of panels with notched edges was 16.4×10^{-3} cubic foot per minute per foot ($1.52 \times 10^{-3} \text{ cu m}/(\text{min})(\text{m})$) of perimeter in the free molecular flow regime.

Offgassing and conditioning of materials. - The offgassing characteristics of the various materials used in the insulation system must be known in order to ensure the self-evacuating capability of the insulation panels when they are installed on cryogenic tankage. These gases, if not condensable or not having a low vapor pressure, could be a major limitation on the vacuum level obtainable in a panel. At liquid-hydrogen temperature, the only gases that exhibit a high vapor pressure are helium, hydrogen, and carbon monoxide.

The materials tested for offgassing included polyurethane-foam spacer materials (both open and closed cell), the radiation-shield aluminized Mylar, and a sample of Mylar laminate casing material with Narmco 7343/7139 adhesive.

The materials were evaluated by using both pumpdown and pressure-rise techniques (ref. 8). This method consists of calculating pressure decay or pressure rise rates based on the pumpdown or settle-out pressure histories of the various materials. In addition, samples of the residual gas after settle out were analyzed.

Open-cell foam did not appear to offgas significantly on settle out after pumpdown to 1×10^{-3} torr ($1.33 \times 10^{-5} \text{ N/cm}^2$). Also a pumpdown for the open-cell foam to this pressure appears to be sufficient for conditioning the foam. After pumping to a lower pressure, backfilling with air to 1 atmosphere (10.13 N/cm^2) for 1 hour, and subsequently re-pumping to 1×10^{-3} torr ($1.33 \times 10^{-5} \text{ N/cm}^2$), no appreciable change was noted in the settle-out pressure. Closed-cell foam had a much higher settle-out pressure and offgassing rate.

The aluminized Mylar radiation shields required preconditioning prior to the pump-down to 1×10^{-3} torr (1.33×10^{-5} N/cm²) by heating in air at 200° F (366° K) for 24 hours. This procedure was necessary to reduce the offgassing rates.

Offgassing tests conducted on a casing material of Mylar laminate with Narmco 7343/7139 adhesive are attributable to condensables, chiefly water, nitrogen, or carbon monoxide; therefore, a pumpdown to 1×10^{-3} torr (1.33×10^{-5} N/cm²) pressure is sufficient. Typical offgassing rates and noncondensable partial pressures for all materials tested are presented in table II.

TABLE II. - SUMMARY OF OFFGASSING TESTS

Material	Experimental total offgassing at room temperature		Calculated total pressure at liquid-hydrogen temperature	
	μ liter/(sec)(ft ²)	μ liter/(sec)(m ²)		
			torr	N/cm ²
Open-cell foam	0.1×10^{-2}	1.08×10^{-2}	4.4×10^{-3}	5.85×10^{-5}
Closed-cell foam	1.0	10.8	4.9	6.52
Aluminized Mylar radiation shields	2.5	26.9	5.3	7.05
Mylar laminate casing material and Narmco adhesive joint	4.2	45.2	4.7	6.25
Empty chamber	---	----	1.3	1.73

As a further conditioning procedure for prefabricated panels, a hydrogen getter (Linde G-2) was used to lower the partial pressure of hydrogen in the panels below 1×10^{-5} torr (1.33×10^{-7} N/cm²).

Full-Scale Insulation-Panel Tests

Representative-size (3- by 4 ft (91.44- by 122-cm)) panels (see fig. 8) were fabricated and installed on a segment-type tank for demonstration testing. This testing was conducted to verify preliminary test results with regard to the sealing capability of the casing material, the low offgassing rates of the materials involved, and the capability of CO₂ gas to cryopump to low panel pressure. Following these tests, panels 6 feet (183 cm) long and 20 to 40 inches (50.8 to 102 cm) wide were fabricated and installed on the 30-inch- (76.2-cm) diameter calorimeter tank to determine the thermal performance of the system for both ground-hold and space conditions.

The unit weight of the installed insulation panels was 0.34 pound per square foot

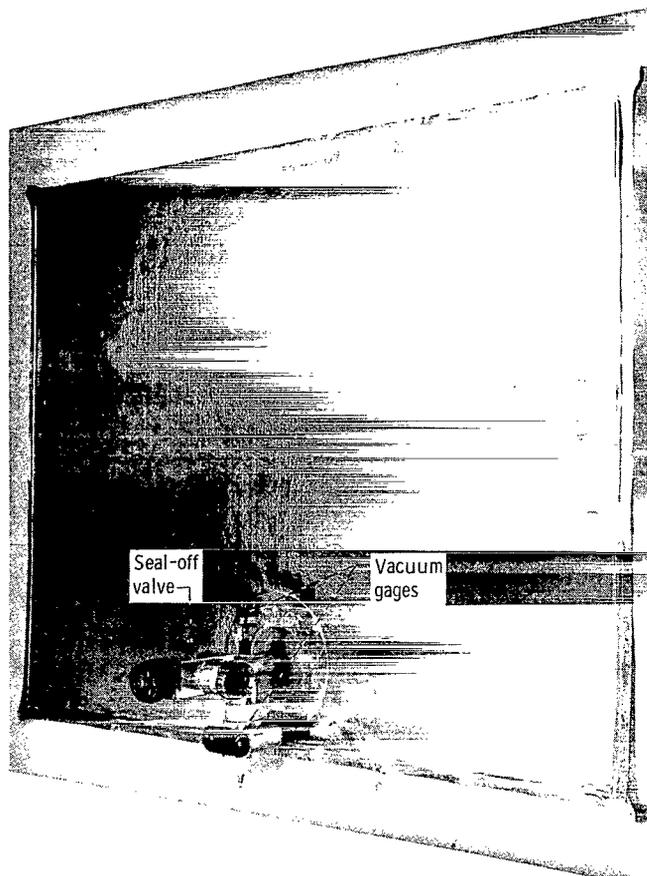


Figure 8. - Insulation panel (3 by 4 ft (91.44 by 122 cm)) for vacuum-seal evaluation.

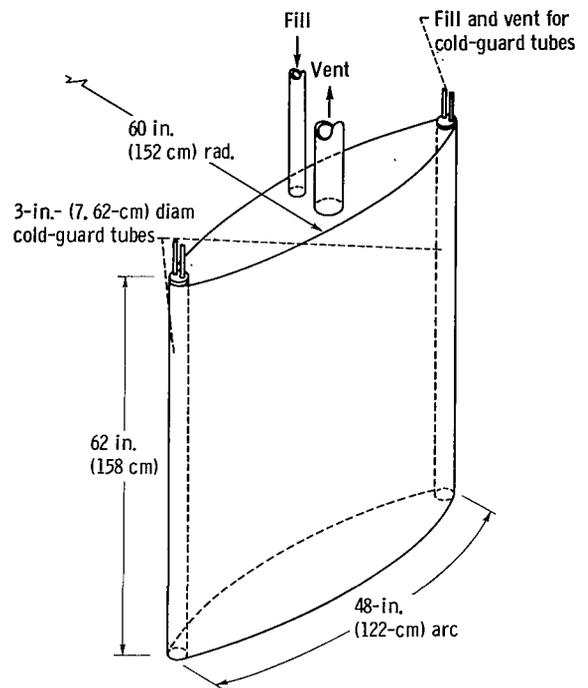


Figure 9. - Design of circular-segment tank used to develop insulation system.

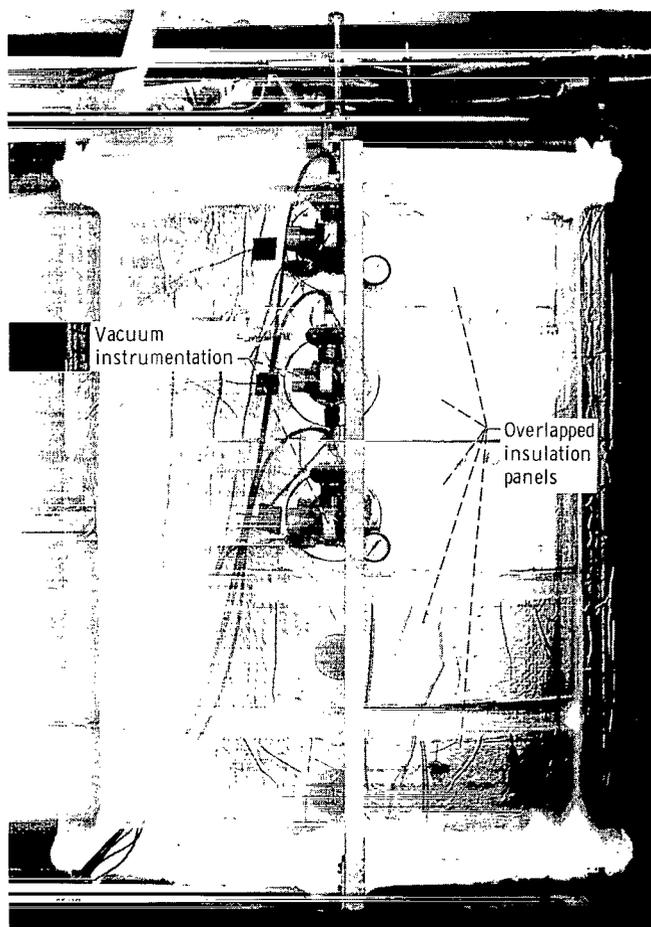
(1.66 kg/m^2) with an 18-radiation-shield multilayer insulation (6 shields per panel) in an overlapping shingle arrangement.

Vacuum seal evaluation of installed panels. - The initial full-size (3- by 4-ft (91.44- by 122-cm)) panel testing was conducted to demonstrate the leak tightness of the vacuum casing material and adhesive joints, and also to demonstrate that the insulation-material offgassing would not cause an insulation-panel pressure rise during an 8-hour liquid-hydrogen test period. A vacuum was obtained within the panels by mechanical pumping. Self-evacuation by cryopumping was not used in order to assure an initial low pressure within the panels when evaluating the vacuum seal.

Panels were fabricated by utilizing four layers of sliced open-cell rigid polyurethane foam separators 0.020 inch (0.0508 cm) thick and three aluminized Mylar radiation shields alternately spaced with the separators. The aluminized Mylar laminate casing material used to enclose the insulating materials was sealed at the panel edges with Narmco 7343/7139 adhesive. Figure 8 shows a full-size demonstration panel fabricated for installation on a segment-type tank. The Narmco adhesive was applied to one side of

the panels in a 6-inch (15.2-cm) spaced, 1-inch (2.54-cm) wide interrupted-grid pattern (gaps in adhesive line to allow cross-gas flow) for attaching to the tank wall and to overlapping panels when installed. A sketch of the segment-type tank is shown in figure 9; it is a small-volume tank with large cylindrical curvature. The panels were installed on the tank in an overlapping shingle arrangement (with only one-third of any panel in direct contact with the tank wall), as described previously and depicted in figures 1 and 2. Figure 10 shows a completely insulated segment tank under test.

After installation on the tank, the panels were leak checked. It was determined that the permeability plus leak rate was about 2.5×10^{-6} cubic centimeters of helium per second per square foot per atmosphere ($2.29 \times 10^{-8} \text{ cm}^3 \text{ He}/(\text{sec})(\text{m}^2)(\text{N}/\text{cm}^2)(\text{at STP})$). This rate compares favorably with that previously determined on small-scale permeability tests (table I). Subsequently, the panels were evacuated for a period of 3 hours and 40 minutes with a diffusion vacuum pump prior to filling the tank with liquid nitrogen for initial cooldown purposes. After an 18-hour cooldown period, during which time the



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Figure 10. - Insulated segment tank filled with liquid hydrogen.

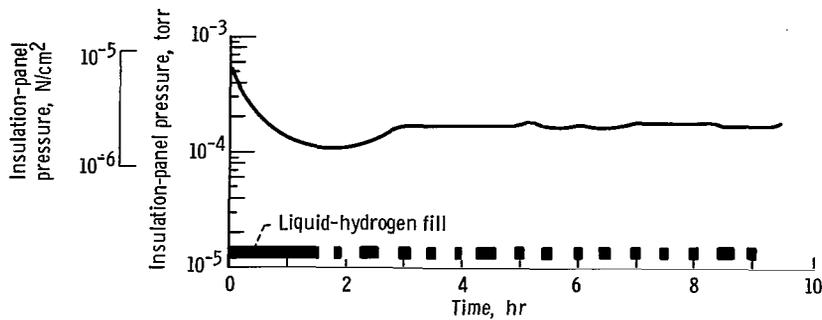


Figure 11. - Pressure history with 3- by 4-foot (91, 44- by 122-cm) insulation panel installed on tank of liquid hydrogen.

vacuum pumps continued to evacuate the panels, the liquid nitrogen was removed from the tank and it was filled with liquid hydrogen. Just prior to the initial liquid-hydrogen fill, the vacuum pumps to the panels were valved off. The panel pressure at that time was about 5×10^{-4} torr (6.65×10^{-6} N/cm²). Figure 11 shows a complete pressure history of a particular panel for the entire $9\frac{1}{2}$ -hour period. Pressures were monitored from the time of the initial liquid-hydrogen fill. As shown in figure 11, except for slight ripples or perturbations between liquid-hydrogen fills, the panel pressure remained essentially constant at about 1.5×10^{-4} torr (1.99×10^{-6} N/cm²) for the entire test period after the initial fill. This test clearly demonstrates that the lightweight flexible casing material bonded with the Narmco adhesive performed satisfactorily in providing a leak-tight enclosure for the basic insulation material. In addition, for these particular test conditions, offgassing of the materials was negligible because no pressure rise was observed over the test period.

During the warmup period following the testing, two of the six panels on the segment tank developed a leak. After removal of these panels from the tank, it was determined that small tears had developed along the edge of the portion of the panel that is bonded directly to the tank wall and immediately below or before the point where the panel overlaps another panel.

Cryopumping capability of carbon-dioxide-filled panels. - Subsequent testing of full-size panels on the segment tank at ground-hold conditions was conducted with liquid hydrogen to determine the panel pressure that would be attainable as a result of self-evacuation by cryopumping when panels are initially charged with CO₂ gas. Fabrication materials and procedures for the new panels of this test were the same as those for the panels of the previous test. Different conditioning was used for each of the panels in this test, however, in an attempt to determine an optimum procedure for conditioning prior to attachment to the tank.

The insulation panels were bonded to the tank and to each other by applying adhesive in only 1-inch- (2.54-cm) wide horizontal strips every 6 inches (15.2 cm) on both surfaces to be bonded in an attempt to preclude panel failure on warmup, as had previously

been experienced. Previously, a 6-inch- (15.2-cm) square gridwork pattern was used in applying adhesive in the initial panels mounted on the segment tank. A 1-inch (2.54-cm) gap was also left in the center of each horizontal adhesive strip to allow for cross-gas flow. The adhesive used in bonding these panels to the tank and to each other was Goodyear G-207 contact adhesive.

After installation on the tank, the panels were evacuated by vacuum pumps to a pressure level of about 10^{-2} torr (1.33×10^{-4} N/cm²) and subsequently backfilled with 1 atmosphere (10.13 N/cm²) of CO₂ gas. This procedure was repeated, with the panel valved off after the final backfill with CO₂ gas. The tank was then initially cooled down with liquid nitrogen for a period of about 22 hours to determine the final pressure with liquid nitrogen. The liquid nitrogen was then removed, and the tank was subsequently filled with liquid hydrogen. Figure 12 shows the complete panel pressure-time history from

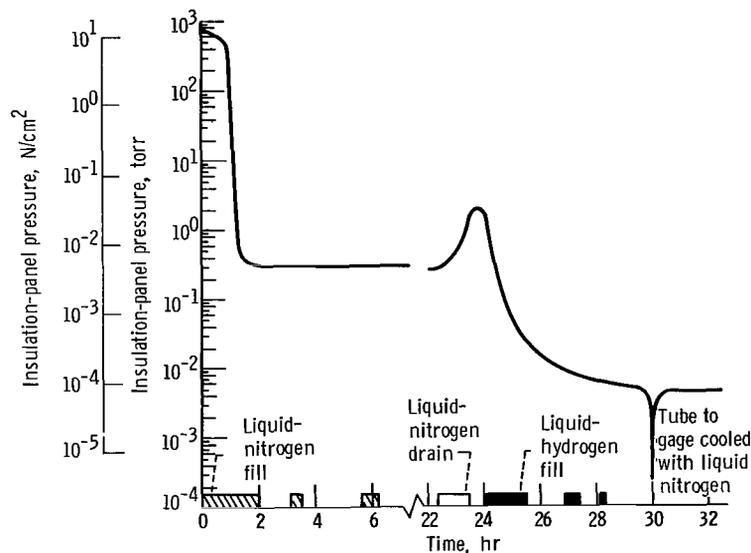


Figure 12. - Self-cryopumped pressure history within 3- by 4-foot (91.44- by 122-cm) insulation panel installed on tank cooled by liquid nitrogen and liquid hydrogen.

the start of the first liquid-nitrogen filling operation. During the liquid-nitrogen fill, the lowest panel pressure level of about 3×10^{-1} torr (3.99×10^{-3} N/cm²) was attained after 2 hours from the start of the initial liquid-nitrogen fill. As expected, the panel pressure rose somewhat during the time period in which the liquid nitrogen was removed and the tank subsequently filled with liquid hydrogen. After maintaining a reasonable level of liquid hydrogen within the tank, further cryopumping decreased the panel pressure to a level of about 5×10^{-3} torr (6.66×10^{-5} N/cm²). This pressure level was attained about 2 hours after the first liquid-hydrogen fill.

Near the end of the 8-hour liquid-hydrogen test period, the panel apparently was in a steady-state condition. The copper tube connecting the vacuum gage with the panel

manifold was cooled to liquid-nitrogen temperature to determine if cryopumpable CO_2 was present in the tube and the panel. With the tube cooled, a pressure of 3.8×10^{-4} torr (5.05×10^{-6} N/cm²) was attained (see fig. 12), indicating that the major portion of the residual gas was CO_2 that had not been cryopumped by the panel.

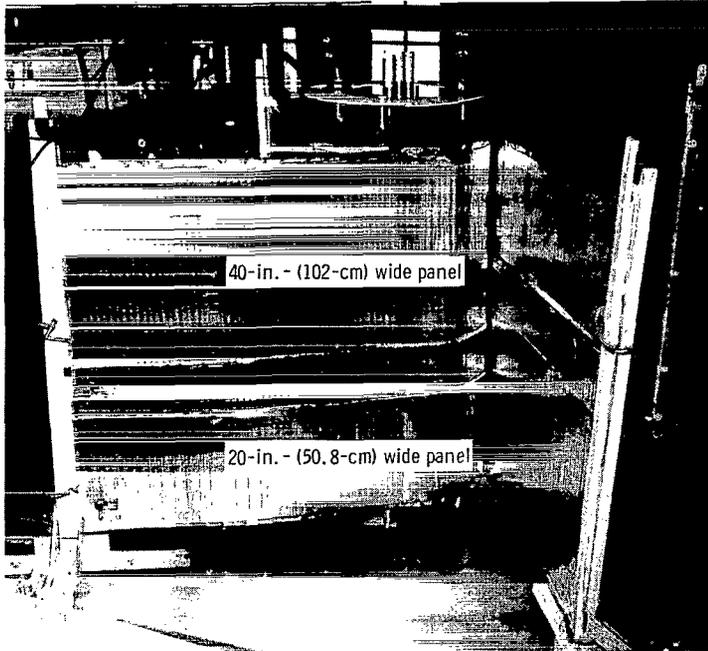
The ultimate pressure 5×10^{-3} torr (6.66×10^{-5} N/cm²) may be caused by poor gas-flow conductance. If the pumping speed were increased by better gas-flow conductance, a lower panel pressure could be achieved and maintained, as demonstrated and discussed in the previous section where a pressure of 1.5×10^{-4} torr (1.99×10^{-6} N/cm²) was maintained for $9\frac{1}{2}$ hours.

Also, an equilibrium pressure of 5×10^{-3} torr (6.66×10^{-5} N/cm²) is not as serious a threat to the insulation thermal performance as might first be thought if it is considered that, when the panels are launched into space, gas permeability into the insulation space will be terminated and a lower panel pressure will result. In addition, the insulation material will become decompressed because of the absence of an atmosphere, and the panel should exhibit a greater pumping speed for the limited amount of gas remaining in the panel.

Insulation-system thermal performance. - The thermal performance of full-size insulation panels (40 by 72 in. (102 by 183 cm) and 20 by 72 in. (50.8 by 183 cm)) was determined on the 30-inch- (76.2-cm) diameter cylindrical calorimeter used previously for the basic multilayer insulation tests. The size of the calorimeter tank prevented use of the vertical shingle arrangement of the panels (fig. 2). Full-length panels in the overlapping direction are necessary to minimize lateral heat flow (solid conduction) along the panels from the outside surfaces to the cold tank walls. It was necessary, therefore, to install full-length panels overlapping around the calorimeter tank circumference rather than along the short vertical axis of the test section. The calorimeter tank being insulated and completely insulated with these panels is shown in figures 13 and 14, respectively.

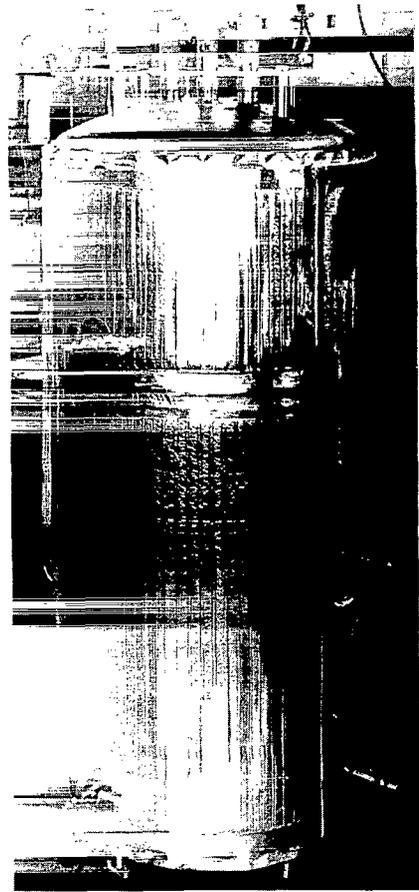
A ground-hold thermal test was performed to determine the system heat flux after self-evacuation which causes a 1-atmosphere (10.13 -N/cm²) compressive pressure on the panels. With the tank side of the shingled panels at liquid-hydrogen temperature and the outer portion of the panels at 70° F (294° K), a heat flux of 23.0 Btu per hour per square foot ($261\ 000$ J/(hr)(m²)) was measured. This heat flux compares favorably with a heat flux of 120 Btu per hour per square foot (1.362×10^6 J/(hr)(m²)) for a ground-hold helium-purged multilayer insulation system (ref. 2) and approximately 35.0 Btu per hour per square foot ($397\ 000$ J/(hr)(m²)) for a sealed-foam insulation system of equivalent unit-area weight (ref. 9).

After the ground-hold thermal test, the calorimeter tank was installed in a vacuum chamber, and the chamber was evacuated to a low pressure to simulate the conditions of space. This lowered pressure relieved the atmospheric compressive pressure on the



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Figure 13. - Installing overlapping insulation panels around circumference of calorimeter tank.



C-66-3136

Figure 14. - Insulation panels installed on calorimeter tank.

panels. The heat flux under self-evacuating space conditions with liquid hydrogen measured 5.1 Btu per hour per square foot ($57\,900\text{ J}/(\text{hr})(\text{m}^2)$) while a skin temperature of 70° F (294° K) was maintained. The pressure in the panels was not measured during this test. However, it can be concluded that the pressure was of the order of 5×10^{-3} torr ($6.66 \times 10^{-5}\text{ N}/\text{cm}^2$) as previously measured in the panels mounted on a segment tank. Figure 6 shows the expected heat flux of an idealized insulation (not considering the casing-material edgewise heat leak) as affected by pressure in the insulation. For a panel pressure of 5×10^{-3} torr ($6.66 \times 10^{-5}\text{ N}/\text{cm}^2$), an idealized insulation would have a heat flux of the order of 3 Btu per hour per square foot ($34\,000\text{ J}/(\text{hr})(\text{m}^2)$).

A thermal test was conducted by opening the panels and mechanically evacuating the entire system through the vacuum chamber in order to determine the insulation performance that could be achieved in the space condition if the panel pressure was below 1×10^{-4} torr ($1.33 \times 10^{-6}\text{ N}/\text{cm}^2$). When this pressure was achieved, as indicated by vacuum gages mounted on the panels, the calorimeter tank was filled with liquid hydrogen.

Within 19 hours the measured heat flux had stabilized at 0.86 Btu per hour per square foot ($97\,700\text{ J}/(\text{hr})(\text{m}^2)$) with the panel outer-surface temperature maintained at 70° F (294° K). This thermal test was continued for 86 hours with no degradation of thermal performance. This excellent thermal performance compares well with the thermal performance of 0.36 Btu per hour per square foot ($4090\text{ J}/(\text{hr})(\text{m}^2)$) achieved with the basic multilayer insulation tested on the calorimeter tank, which did not have any heat-flux contribution by lateral thermal conduction of a vacuum casing material.

The improved performance realized when the panel is at the lower internal pressure of 1×10^{-4} torr ($1.33\times 10^{-6}\text{ N}/\text{cm}^2$) indicates that a redesigned system which would provide for a higher gas-flow conductance and thus a lower self-evacuated pressure would produce better thermal performance in space. An alternate redesign for assuring a low panel pressure would be to design the panels so that they could be mechanically opened while in space; thereby, the multilayer insulation would be evacuated by using the vacuum of space.

SUMMARY OF RESULTS

The results of this investigation to develop and evaluate a self-evacuated multilayer insulation of lightweight prefabricated panels for cryogenic space propulsion vehicles can be summarized as follows:

1. It is feasible to use, for both ground-hold and space storage of cryogenic propellants, a lightweight evacuated multilayer insulation of 18 radiation shields assembled in prefabricated panels that weigh only 0.34 pound per square foot ($1.66\text{ kg}/\text{sq m}$) installed. The insulation panels are mounted on cylindrical tanks in an overlapping shingle arrangement.
2. Self-evacuation by cryopumping of multilayer insulation panels charged with carbon dioxide gas installed in a shingle arrangement on a tank containing liquid hydrogen was achieved. Pressures as low as 5×10^{-3} torr ($6.66\times 10^{-5}\text{ N}/\text{cm}^2$) were measured.
3. Open-cell polyurethane foam multilayer separators between radiation shields exhibit the lowest heat transfer under compressive pressure of the many materials tested. The successful use of this foam sliced to only 0.02 inch (0.0508 cm) thick contributed significantly to the effective thermal performance of the insulation system, particularly under compressive pressures below 1 atmosphere ($10.13\text{ N}/\text{cm}^2$).
4. Thermal performance of the insulation system installed on a 30-inch- (76.2-cm) diameter calorimeter tank containing liquid hydrogen and with a panel surface temperature of 70° F (294° K) was measured as follows:
 - a. For ground-hold tests with self-evacuated panels, the heat flux was determined to be no greater than 23 Btu per hour per square foot ($261\,000\text{ J}/(\text{hr})(\text{m}^2)$). This

heat flux is about one-fourth of that obtained by helium-purged multilayer insulation and is somewhat less than that for sealed-foam insulation of equivalent unit weight.

b. For simulated space tests in a vacuum chamber, the heat flux with self-evacuated panels was determined to be 5.1 Btu per hour per square foot (57 900 J/(hr)(m²)). This somewhat high heat flux was attributed to high panel gas pressure caused by poor gas-flow conductance.

c. For simulated space tests in which the panel gas pressure was reduced by mechanical pumping, the heat flux was determined to be 0.86 Btu per hour per square foot (97 700 J/(hr)(m²)). This heat flux compares favorably with the 0.36 Btu per hour per square foot (4090 J/(hr)(m²)) that was measured for basic multilayer insulation which did not have a heat-flux contribution from lateral conduction of a vacuum casing.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 11, 1967,
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