99

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DEVELOPMENT OF METHODS FOR APPLICATION OF POLYURETHANE SPRAY FOAM INSULATION SYSTEMS TO LIQUID HYDROGEN TANKS

By James M. Carter
Manufacturing Engineering Laboratory

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Marshall Space Flight Center, Alabama

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MECHANICAL AND CHEMICAL DEVELOPMENT BRANCH
MANUFACTURING RESEARCH AND TECHNOLOGY DIVISION
MANUFACTURING ENGINEERING LABORATORY

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ABSTRACT

Presented in this report are the methods for insulation of a liquid hydrogen container with polyurethane spray foam. The spray foam equipment and its operation; the methods and techniques for application of spray foam to the surface of the tank; and the sealing of the external surface of the foam are described.

Several test items which were insulated with polyurethane spray foam and general results of cryogenic testing of the insulation system are discussed.

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TABLE OF CONTENTS

TABLE OF CONTENTS (Cont'd)

LIST OF ILLUSTRATIONS

SECTION III.

SECTION IV.

SECTION V.

SECTION VI.

DEVELOPMENT OF METHODS FOR APPLICATION OF POLYURETHANE SPRAY FOAM INSULATION SYSTEMS TO LIQUID HYDROGEN TANKS

SUMMARY

The various types of equipment that are available for application of polyurethane spray foam for cryogenic insulation are presented. The Binks "Hot Hydraulic" Spray Foam Formulator is discussed in detail along with modification to the formulator which have been made by S&E-ME.

Methods for insulation of cylindrical vehicles with polyurethane spray foam, including surface preparation, foam application, and surface finishing are covered.

Several test items which have been insulated by S&E-ME with polyurethane spray foam and the general results of cryogenic testing of the insulation systems are discussed.

SECTION I. INTRODUCTION

Polyurethane Foam has been used for several years as an insulation for LH_2 storage tanks and as a lightweight filler material in the S-IC fuel tank. In both instances, the materials used were slow reacting pour foams which produced a foam having quite variable cell size and shape. These foams have usually been somewhat brittle although the performance of the foam was reasonably satisfactory.

Early in 1967 there were two applications proposed for LH_2 container insulation on large flight type structures.

The Mechanical and Chemical Development Branch of the Manufacturing Research and Technology Division proposed the use of spray or pour foam for the insulation system of the Nuclear Ground Test Module and independently North American/Rockwell proposed spray foam as the insulation for the S-II vehicle. Both proposals were predicated upon the physical and mechanical properties of recently developed polyurethane spray foams; and, an excellent probability of success along with improved thermal performance and better manufacturing characteristics was indicated.

SECTION II. REQUIREMENTS FOR A POLYURETHANE SPRAY FOAM INSULATION SYSTEM

The two insulation systems had many common requirements and were both to be applied to vehicles approximately the same size; 33-feet in diameter and more than 40 feet in length.

The requirements for use of this type insulation are reasonably easy to state but somewhat difficult to nondestructively prove or even technically define.

A. GENERAL REQUIREMENTS FOR NUCLEAR GROUND TEST MODULE INSULATION SYSTEM

The general requirements of the insulated system for the Nuclear Ground Test Module are:

1. Low K factor, less than 0.32 $Btu/ft^2\ {}^oF/in$
2. Stay in place during an extended test period.
3. Be sufficiently radiation resistant to complete the test period.
4. Be easily applied.
5. Be easily protected from atmospheric exposure.

B. GENERAL REQUIREMENTS FOR S-II INSULATION SYSTEM

The general requirements for S-II application are:

1. Less than 0.75K factor (Btu/ft.^{2} °F in.)
2. Stay on the vehicle during tanking with LH_2 and during flight.
3. Be white enough for tracking in flight.
4. Be lightweight.
5. Be reasonably easy to apply.
6. Be easily protected from atmospheric exposure.

In both cases the insulating value of polyurethane spray foam significantly exceeded the required value, i.e., the room temperature K factor for polyurethane foam is 0.12 BTU/ft.^{2} °F/in.

Weight consideration for the S-II stage is quite important and the results of using the 2 lb./ft.^{3} density foam to replace the then used honeycomb and tedlar face sheet sandwich insulation (commonly called 1.6 insulation) resulted in a saving of weight exceeding 2,400 lbs. per vehicle, and has eliminated purge requirements which were costly, time consuming, and at times, disastrous.

In order to survive S-II test and flight conditions, the foam must withstand both flight forces and atmospheric pressure force of 15 psi due to cryopumping near the tank-insulation interface. Test results show room temperature strengths of approximately 40 psi in compression and about 58 psi average tensile strengths with a Young's modulus of 2500-4000 psi (P.T.) Cryogenic temperature increases the modulus to about 8000 psi with very little apparent change in tensile strength; therefore, the foam insulation system could be expected to survive both flight and atmospheric requirements since flight requirements are on the order of 10 psi.

There are other environments such as temperature and vacuum encountered during the flight of the S-II which must also be overcome for the one time exposure. Tests to date indicate that the selected materials accept exposure to vacuum, and temperature up to 550°F for short periods with minimum insulating value loss and little ablation of the surface thereby adequately satisfying the flight profile exposure requirements. However, the foam is not the required color initially, and turns a tan-yellow when exposed to sunlight. This color change is accompanied by a surface degradation which appears as a powdering effect. These conditions can be prevented by application of an opaque coating. This necessitates the use of a covering which is easily applied; is vapor and air tight; and either comes off in small drops or stays intact during flight.

The selected coating is a white pigmented polyurethane compound followed by a surface coat of Dynatherm. The combination has reacted consistently, to date, producing small drops which wipe away in the slip stream. Tests on the X-15 Aircraft (NASA) bear out this result in a documentary film made during high altitude flight simulating the S-II trajectory.

No flight requirements existed for the Nuclear Ground Test Module and the proposed foam insulation appeared to meet all requirements.

In order to support both of these programs, the Manufacturing Engineering Laboratory developed the capability for application of polyurethane spray foam insulation systems.

SECTION III. SPRAY FOAM APPLICATION EQUIPMENT

Currently available polyurethane spray foam systems generally consist of an isocyanate prepolymer called the "A" component and the polyol called the "B" component. The "B" component normally contains the calalyst, the blowing agent, the surfactant, and the fire retardant. The polyol, assisted by the catalysts, reacts with the isocyanate to initiate an exothermic reaction; during the reaction the heat generated vaporizes the blowing agent causing the formation of cells; and the surfactant causes the cells to be uniform in size and shape.

The function of the foam application equipment is to meter a definite amount of each component such that a stoichemetric reaction can occur between the components when they are mixed in a mixing chamber.

The rate of reaction required to insure application of foam to vertical surfaces without excessive sagging is such that the mixed components must have an extremely short dwell time in the mixing chamber, or the reacted materials will polymerize in the chamber, creating an excessive time delay while the equipment is cleaned.

A. REQUIRED FUNCTIONS OF APPLICATION EQUIPMENT

Because of the basic problems inherent in handling polyurethane foams, the following requirements must be met in order to insure a reliable piece of application equipment:

1. The spray head and hose bundle must be light enough for easy positioning and hand operation without unduly tiring the operator.
2. The mixing system must be capable of complete mixing at all output levels for long periods of operation.
3. The mix chamber cleaning system should clean the mix chamber well enough to allow several hours between purge and spray initiation without detrimental effects.

4. The proportioning system must dispense the components to be mixed in a continuously metered flow within the allowable variation in mix ratio normally allowed (+ 1 percent) by the material manufacturer.
5. The spray equipment must apply the foam in a manner so that a reasonably smooth surface is formed to prevent void inclusions and generate a uniform cell structure throughout the foam with a minimum of large and elongated cells.
6. The equipment should be made so that a minimum likelihood of operator error can be expected, i.e., ratio adjustment, ratio measurement, etc.
7. The equipment should be easily maintained during operation and easily cleaned in case of stoppage, i.e., failure to flush after use.

B. TYPES OF APPLICATION EQUIPMENT

Spray foam application equipment commercially available can be classified into two different types, either an air atomized system or an airless system. In either case the equipment used to mix and dispense the foam components consists of a material feed system and a formulator or proportioning unit that is connected by hoses to a spray head. The proportioning unit consists of two pumps which meter the components and supplies the proper amount of each component to the spray head where they are mixed. The mixed, reacting components are expelled from the head, as an atomized spray, with sufficient force to propel the spray onto the surface being foamed.

1. Air Atomized Spray Equipment. An air atomized foam spray unit utilizes an air driven motor in the spray head to mechanically mix the two components. Compressed air is injected into the mixed components for the purpose of atomizing the mixture.

Most of the air atomized spray units have a variable ratio output and can be adjusted to accommodate the particular foam system in use. This is accomplished by either varying the length of one of the pump strokes when using piston type pumps, or by varying the rotational rate of one pump when using gear type pumps.

Foam spray units utilizing this principle of operation usually have output pressures in the range 400 to 700 psi.

2. Airless Spray Equipment. An airless foam spray unit utilizes the hydraulic pressure of the foam components to mix the components and to atomize the mixture.

Most available airless spray units have a fixed ratio output; normally 1:1 by volume, which cannot be changed without a major change to the proportioning unit (changing the internal volume of one of the pumps).

Foam spray units utilizing this principle of operation usually have output pressures in the range of 1500 to 4000 psi.

C. AVAILABLE SPRAY FOAM APPLICATION EQUIPMENT

Several different kinds of application equipment have been obtained for use in the foam development program. By having these different types of equipment, new manufacturing methods and techniques can be determined, as well as duplicating other existing application methods as desired.

1. Lemco Model 510 Foam Unit. This unit, manufactured by the Lake Erie Machine Company, Toledo, Ohio, is a mechanically mixed, air atomized unit in which the ratio between the components and the output of the unit can be varied up to 30 lb./min. The formulator is shown in Figure 1 and the spray head is shown in Figure 2. The unit requires a minimum of 27 cfm. air at 90 psig for spray requirements.

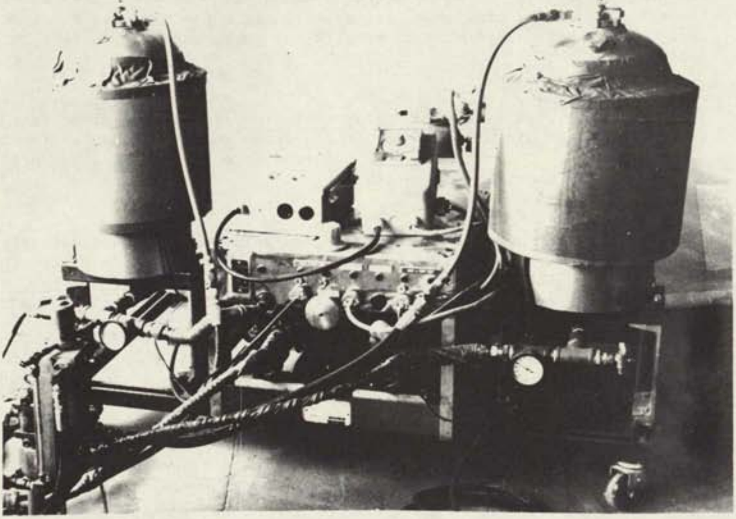


FIGURE I. LEMCO MODEL 510 FORMULATOR

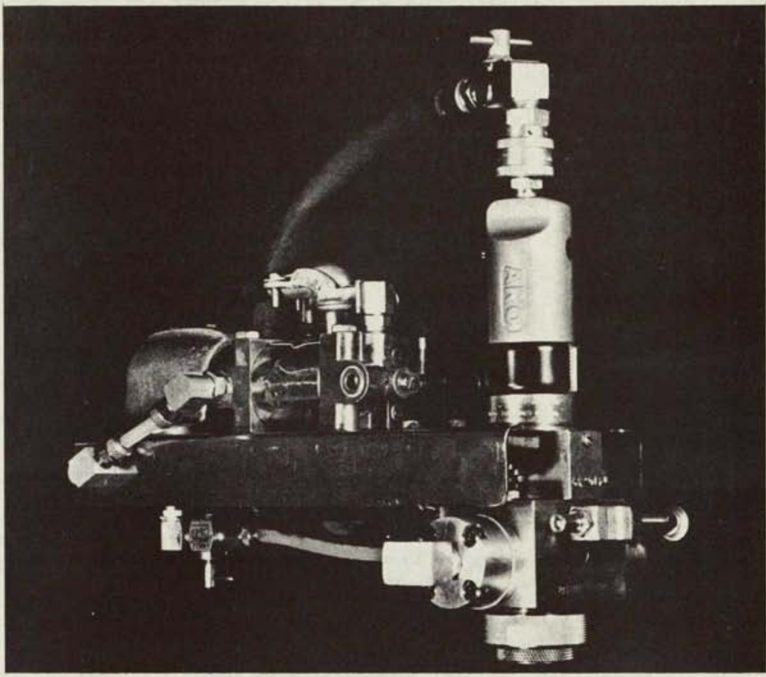


FIGURE 2. LEMCO MODEL FOUR-TEN MIXING HEAD

The Lemco formulator does an excellent mixing job, has a very efficient flush system and, for a mechanical mixed system, is reasonably easy to clean up. The unit circulates the foam components when the spray head is not activated and if one component should be used up before the other, the absence of check valves in the system allows the empty hose to be filled with the wrong component necessitating a lengthy clean up period. Hand spraying operations are very difficult due to the size and weight of the spray head.

2. <u>Glas-Craft Foam Unit</u>. This unit, manufactured by Glas-Craft of California, Glendale, California, also, is a mechanically mixed, air atomized system. The Ratio-Master formulator is shown in Figure 3 and the Sidewinder gun used in conjunction with the formulator is shown in Figure 4.

The RATIO-MASTER is a two component resin pumping system that meters and pumps accurately. The pneumatically driven, positive displacement pumps meter the amount of each component used. A variable pump is utilized on one side of the system enabling ratios of 1-1 to 8-1 to be attained. Two 5 gallon containers hold resins and the machine is equipped with an automatic purging system. Up to 8 lbs. of metered and mixed two-component resin systems may be either sprayed or poured from this unit.

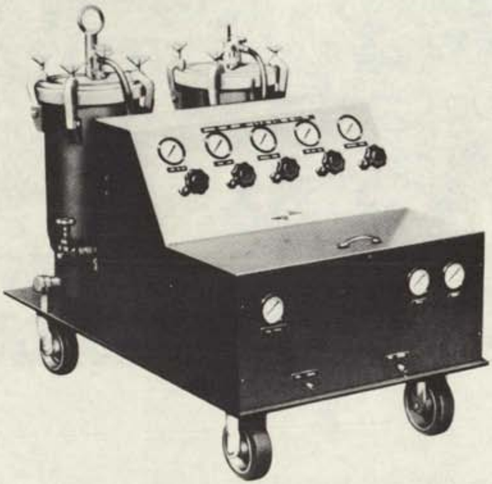


FIGURE 3. GLAS-CRAFT RATIO-MASTER FOAM METERING SYSTEM

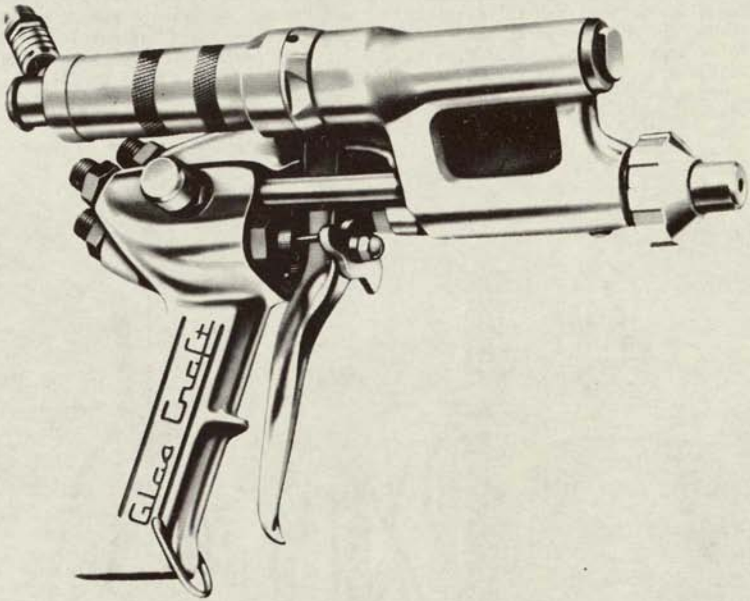


FIGURE 4.   GLAS-CRAFT SIDEWINDER FOAM SPRAY GUN

The system performs a satisfactory job of mixing and dispensing foam, but leaves much to be desired in flushing and cleaning the spray gun since the path through the mix area is rather long and conducive to stoppage from extremely fast reacting material. Also the pumps used in the formulator are not capable of pumping low viscosity materials which precludes its use in outer coating application.

3. <u>Binks "Hot Hydraulic Unit"</u>. This unit, manufactured by Binks Manufacturing Company, Chicago, Ill., utilizes an airless spray method to dispense the foam in which the "A" and "B" components are pumped separately to the airless spray gun where the two components are intimately mixed under pressures which may be as high as 2000 to 3000 psi (13 789 514.4 to 20 684 271.6 N/m^2). Atomization of the mixed components occurs when the fluid is forced through a small orifice in the spray gun tip. The formulator is shown in Figure 5 and the spray gun in Figure 6.

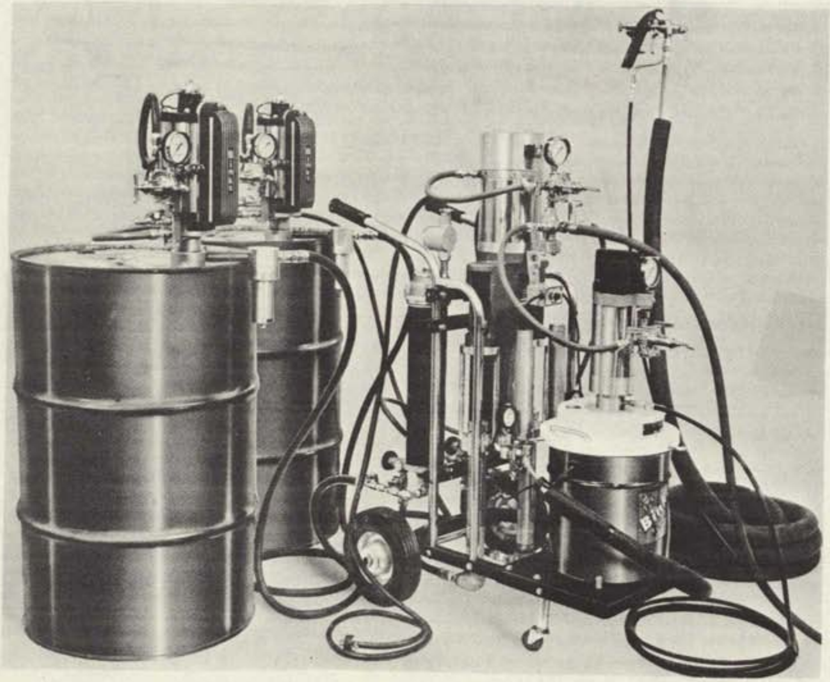


FIGURE 5. BINKS "HOT HYDRAULIC" SPRAY FOAM SYSTEM

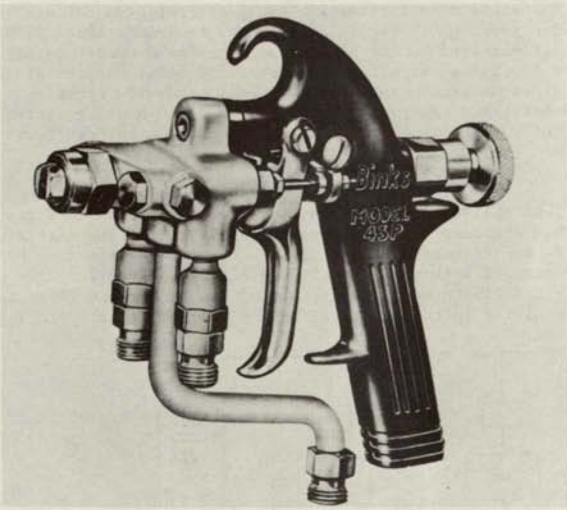


FIGURE 6. BINKS 43P AIRLESS FOAM SPRAY GUN

This system heats either or both of the foam components so that when the components are mixed in the gun a faster reaction will occur, also the viscosity of the two components is lowered, allowing better atomization. This unit was designed to operate at low pressures (500-700psi). The mixing ratio between the foam components is not variable, limiting the unit to application of 1 to 1 ratio foam systems only. The output of the unit can be varied either by changing the pressure of the input air to the formulator or by use of a larger or smaller nozzle on the spray gun.

D. PROCESS EQUIPMENT

The basic foam application equipment selected for use by the Manufacturing Engineering Laboratory at MSFC was the Model 105-1027 Hot Hydraulic Spray Outfit marketed by the Binks Manufacturing Company, Chicago, Ill. shown in Figure 5. Hereafter all part numbers and model numbers are designation of the Binks Manufacturing Company unless otherwise specified. This spray unit consists of a material feed system, a resin heating system, a proportioning unit, a fluid manifold, a spray gun with a solvent flush system, and a cart.

When the Hot Hydraulic Unit was initially used to spray foam it was found that the feed system was not supplying sufficient material to keep the liquid and foam cavitating. This situation was remedied by locating a surge chamber between the resin heater and the suction side of the liquid end. To facilitate maintenance, some of the valves and fluid lines were changed resulting in the spray unit shown diagrammatically in Figure 7.

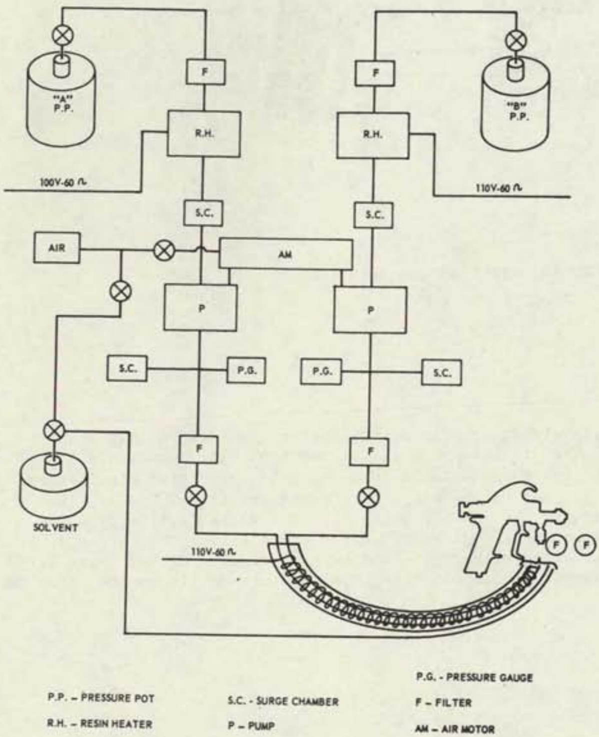


FIGURE 7. MODIFIED HOT HYDRAULIC SPRAY FOAM SYSTEM

1. Material Feed System. The purpose of the material feed system is to provide a constant supply of each foam component to the suction end of the proportioning pumps. This is accomplished by either of the two following methods.

a. 4:1 Ratio Bung Pump Feed. The Lark series bung pump #103-1005 enables materials to be pumped directly from the original 55 gallon shipping containers to the proportioning unit. This pump, shown in Figure 8, is an air operated, positive displacement type pump which consists of an air motor, an air valve, and a fluid section mounted in a standard 55 gallon drum, and supplies fluid at four times the pressure of the operating air.

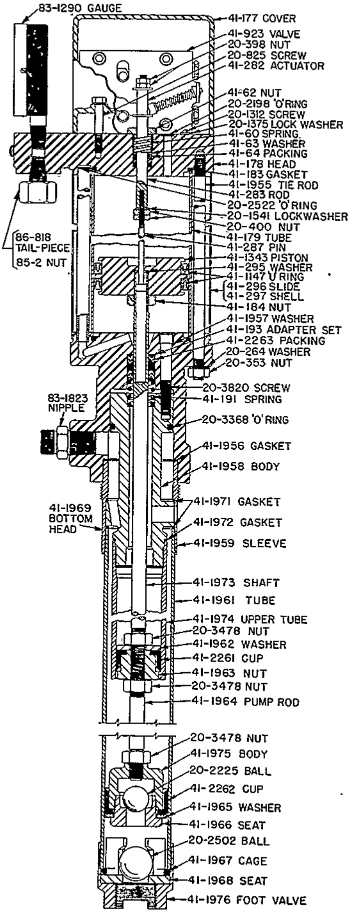


FIGURE 8. MODEL 103-1003. 4 to 1 RATIO BUNG PUMP

It can be noted from Figure 9 that the foot ball valve in the fluid section of the pump depends upon gravity to close the valve. This gives rise, when pumping viscous materials, to the possibility of pump cavitation, causing an interruption in material flow to the suction end of the proportioning pump. When the proportioning pump cavitates, less of the particular material will be metered than is required for complete reaction of the components. This results in a momentary ratio variation which produces a sub-standard area in the resulting foam. These pumps have, however, been used to satisfactorily pump foam components whose viscosities are less than 1000 centipoise.

b. Pressure Pot Feed. Two standard pressure pots, fabricated and tested to ASTM standards can be used for the material feed system. The pots, shown in Figure 9, are mechanically stirred by an air motor, which is connected to an agitator, thereby eliminating the possibility of stratification of either of the foam components.

After leak testing, the pots are filled with the foam components and pressurized with dry nitrogen to a pressure of 60 psi. This will assure a constant supply of the foam materials to the proportioning pumps.

2. Resin Heating System. The purpose of the resin heating system is to heat either or both of the foam components so that they are supplied to the proportioning pumps at a predetermined temperature. The lower viscosity of the components, due to heating, enables them to be pumped more easily to the mixing chamber. This heater is shown in Figure 10.

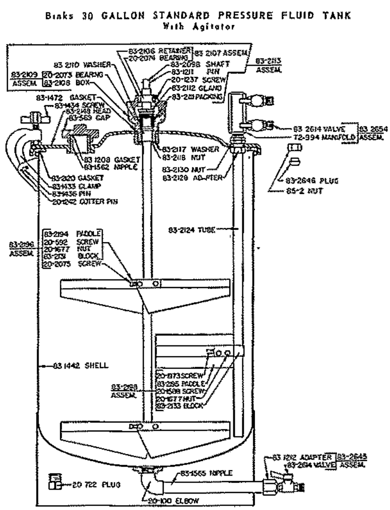


FIGURE 9. STANDARD PRESSURE FLUID TANK

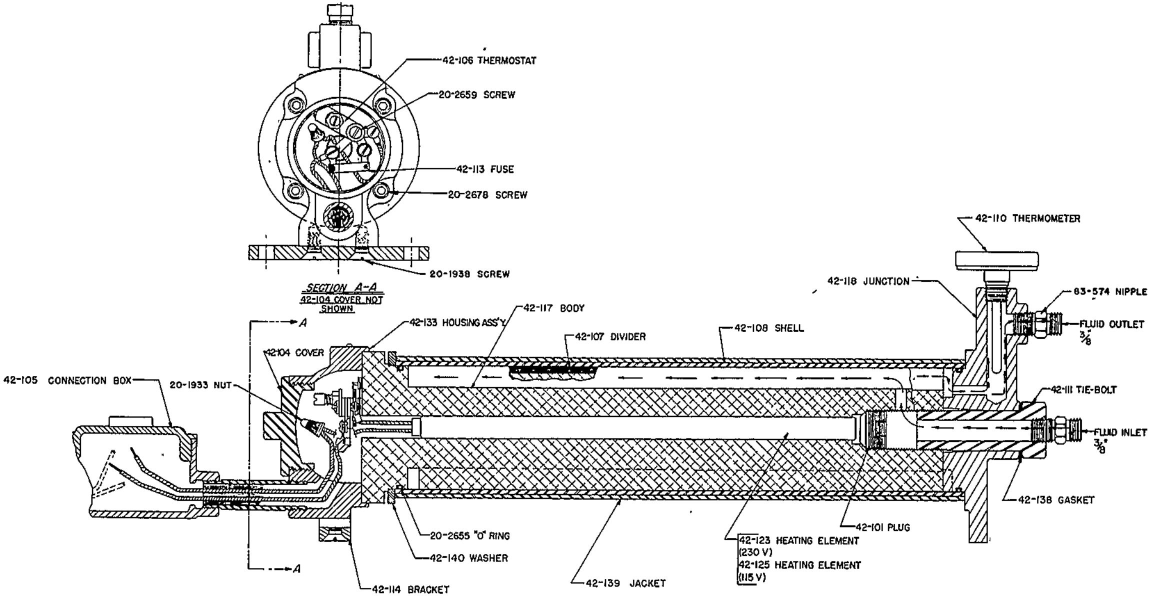


FIGURE 10. MODEL 42-2001 (115 VOLT) RESIN HEATER

3. <u>Proportioning Unit</u>. The function of the proportioning unit is to accurately measure and dispense foam components so that the proper ratio between the components will exist when they are mixed. The proportioning unit of the "Hot Hydraulic Formulator" consist of an air motor and an air valve, two positive displacement piston pumps, and the mounting bracketry to connect the two pumps and the air motor. The pumps are physically tied together through a yoke which is driven up and down by the air motor. The "A" pump is identical to the "B" pump and, when operating, the output ratio between the two pumps is 1:1 by volume.

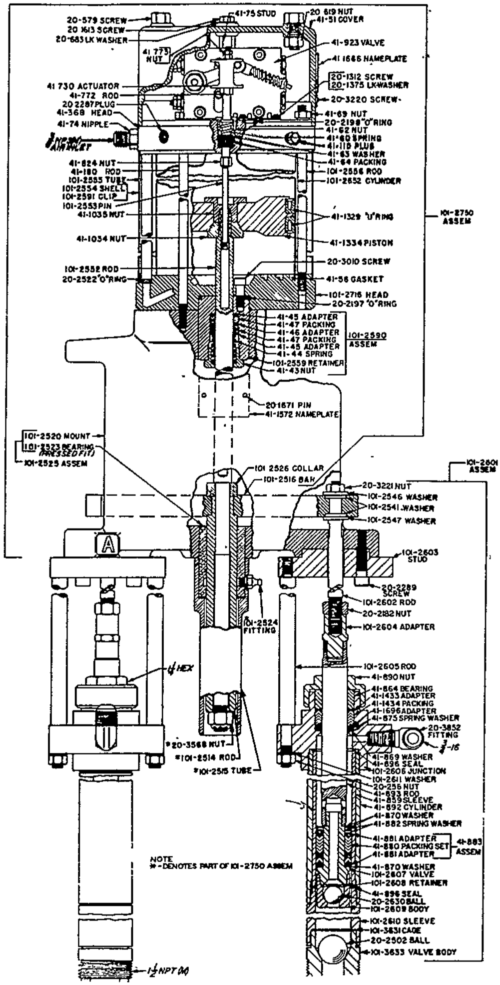


FIGURE 11. MODEL 101-2511 PROPORTIONING UNIT

a. 101-2601 Liquid End. The 105-1027 spray unit is equipped with the 101-2511 Pump Assembly shown in Figure 13. The output of the 101-2601 pumps shown in Figure 11 is 2.2 liquid oz. per stroke. When using shop air (100-115 psi) to drive the 101-2750 air motor (5 in. cylinder) the output pressure of the unit is in the range, 1200-1400 psi. This pump is shown in greater detail in Figure 12.

When determining the operating characteristics of the equipment it was noted that, at the instant the pump changed direction at either the top or bottom of the stroke, the output pressure would fluctuate. This fluctuation can be reduced, but not eliminated, by a slight modification to the pump. This modification consists of locating a spring between the 20-2502 ball and the 101-3631 cage so that the ball will be more rapidly returned to its seat when the valve begins to close.

b. 106-1105 and 106-1106 Liquid Ends. The first foam system proposed for use by North American Rockwell was not a 1.0:1.0 ratio by volume system but was a 1.0:1.4 B/A ratio by volume. Liquid ends were available from Binks Manufacturing Company that permitted the Hot Hydraulic Formulator to mix and apply this non-1:1 ratio foam. The 106-1105, Special 1.0 Liquid End shown in Figure 13 and the 106-1106, Special 1.4 Liquid End shown in Figure 14 were located on the proportioning unit in the "A" and "B" position respectively. These pumps were modified in a manner similar to the 101-2601 Liquid End (See Figure 13 and 14) in that a spring was positioned between the 20-2631 Ball and the 41-1292 Retainer of the 1.0 Liquid End and the 20-2631 Ball and the 101-2598 Retainer of the 1.4 Liquid End.

Shortly after the Hot Hydraulic unit was equipped to spray the 1.0:1.4 ratio foam that foam system was dropped from contention as a candidate system and no requirement existed for spray foam at that particular ratio.

In an attempt to reduce the pulsation at the end of the pump strokes the 2.2 oz. liquid ends were changed to the 1.4 liquid ends which pumps 1.4 fluid oz./stroke/pump. The use of the lower volume pump permits the formulator to be operated at higher output pressures (2200 to 2800 psi). The pumps make more strokes/unit time resulting in less elapsed time at the end of the pump stroke than would elapse at the end of a 2.2 oz. pump stroke. This was observable as a definite decrease in pulsations when using the 1.4 oz. liquid ends to the point where pulsations were not a problem.

Two 106-1105 liquid ends, which pump 1.0 fluid oz./stroke, can also be used on the proportioning unit causing the output pressure of the unit to rise to approximately 3000-3500 psi, as with the 1.4 oz. pumps, pulsation at the end of a stroke were not of such magnitude as to represent a problem.

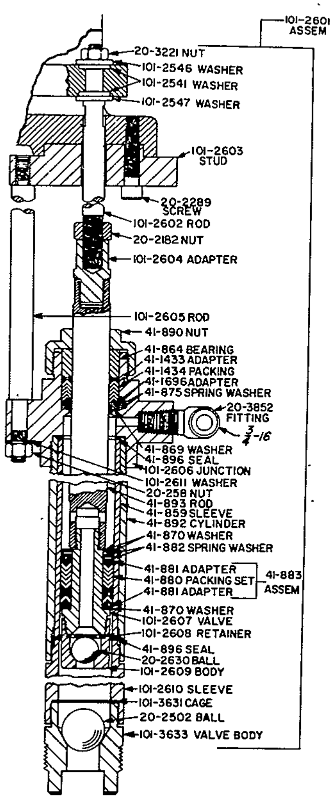


FIGURE 12. MODEL 101-2601 LIQUID END

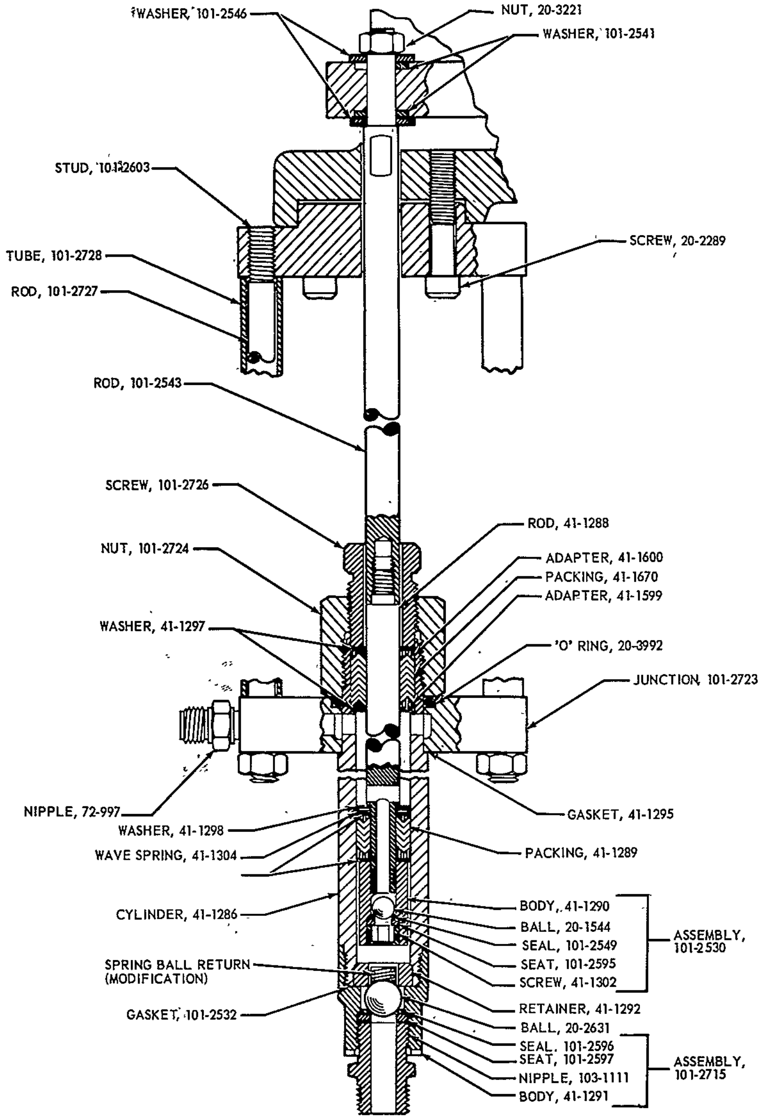


FIGURE 13. MODEL 106-1105, SPECIAL 1.0 LIQUID END

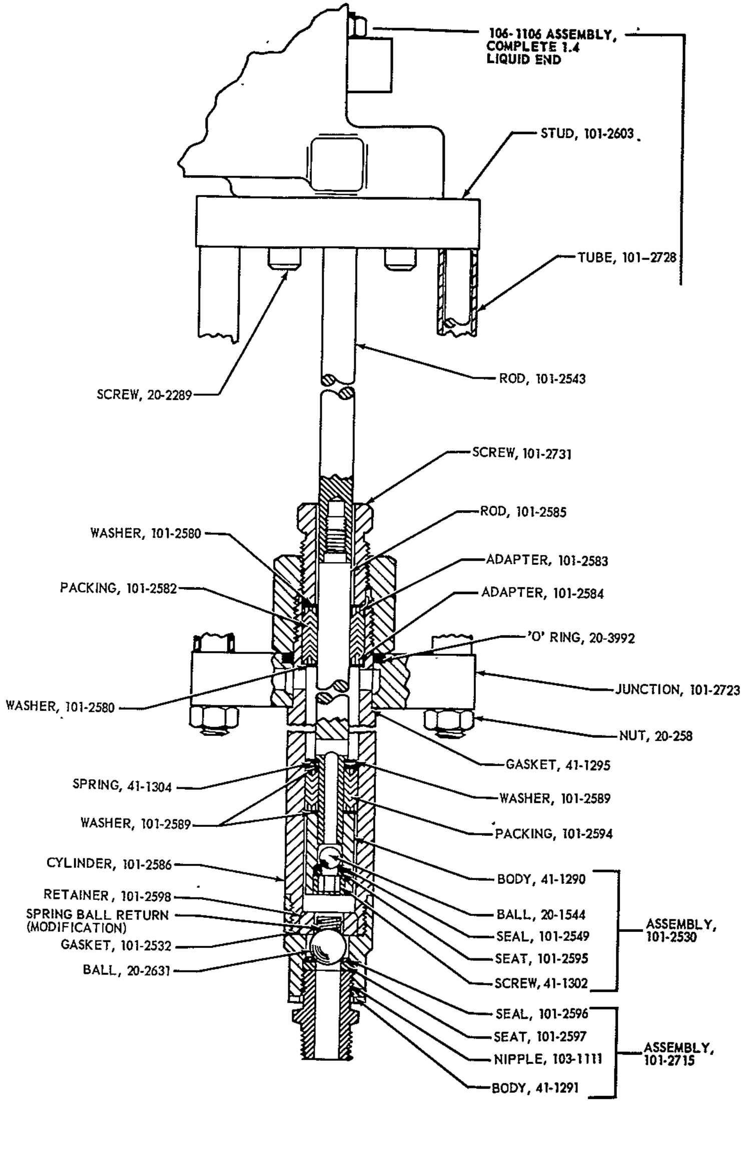


FIGURE 14. MODEL 106-1106, SPECIAL 1.4 LIQUID END

c. Modified 106-1105 Liquid End. After efforts to spray the 1.4 ratio foam were discontinued, a new type foam was obtained (Nopco BX 250A) which had a stoichiometric ratio of 1.12g "A" to 1.09 "B". The Hot Hydraulic formulator was unable to meter this ratio using any of the available combination of pump sizes.

A method of modifying the 106-1105 and 106-1106 pumps was determined. Using the 106-1105 pump as the reference, a cylinder diameter was calculated so that the output volume would be 1.12 times as great in order to maintain equivalent output on both the up and down stroke of the pump, the ratio between cylinder diameter and piston diameter was maintained.

Certain parts from each of the pumps required modification. Parts that had to be altered and the required operations are as follows:

From the 106-1105 Liquid End

1. 41-1286, Cylinder-Machine from 0.750 inch ID to 0.806 inch ID.
2. 41-1599, Adapter-Machine ID of adapter to actual diameter of rod less 0.003 inch.
3. 41-1600, Adapter-Machine ID of adapter to 0.003 inch less than the actual rod diameter.
4. 41-1670, Packing-Machine ID of packing to 0.003 inch less than the actual rod diameter.

From the 106-1106 Liquid End

1. 101-2585, Rod-Machine from 0.592 inch diameter to 0.538 inch diameter.
2. 101-2589, Washer- (bottom washer) Machine ID of washer to 0.015 inch less than packing diameter.
3. 101-2594, Packing-Machine ID of packing to 0.003 inch greater than actual cylinder diameter.

All other parts were from a standard 106-1105 liquid end.

The modified pump was assembled and placed on the "B" side of a proportioning unit with a 106-1105 liquid end on the "A" side of the unit. Check-out of the system showed that the unit dispensed foam in the ratio 1 part "A" to 1.123 parts "B" by weight (average of 5 determinations with a spread of 0.003-0.002). A second pump was similarly modified and upon verification of the unit it was found that foam was dispensed in the ratio 1 part "A" to 1.126 part "B" by weight.

The various liquid ends available for use on the Hot Hydraulic proportioning unit are tabulated in Table 1.

Table 1. Liquid Ends Used On "Hot Hydraulic" Proportioning Unit

Nomenclature	Cylinder Diameter (Inch)	Rod Diameter (Inch)	Output/stroke (Ounce)
101-2601	1.255	0.875	2.2
106-1105	0.750	0.500	1.0
106-1106	0.885	0.594	1.4
106-1105 Mod.	0.806	0.538	1.12

NOTE: Liquid ends are used in pairs to produce a 1:1 ratio by volume between foam components. The 106-1105 Mod. liquid end meters foam components in ratio 1.0:1.12 by weight when used with a standard 106-1105 pump on the "A" side.

4. <u>Fluid Manifold and Output Hose Assembly</u>. The manifold shown in Figure 15, and the hose assembly, shown in Figure 16 provide the connection between the proportioning unit and the spray gun.

The manifold system consists of the manifold, a 2 X 2 X 2 inch aluminum block which is drilled and tapped to accommodate the other elements of the system; a 0 to 5000 psi pressure gauge, a surge chamber, a high pressure filter and drain valve, and a valve to isolate the hose from the manifold and proportioning unit.

The hose assembly consists of two braided stainless steel, teflon lined; high pressure hoses; an electric heater tape; tubular rubber insulation and the electric heater control. The heater tape and control provide a means of warming the foam components to a desired temperature prior to mixing the components.

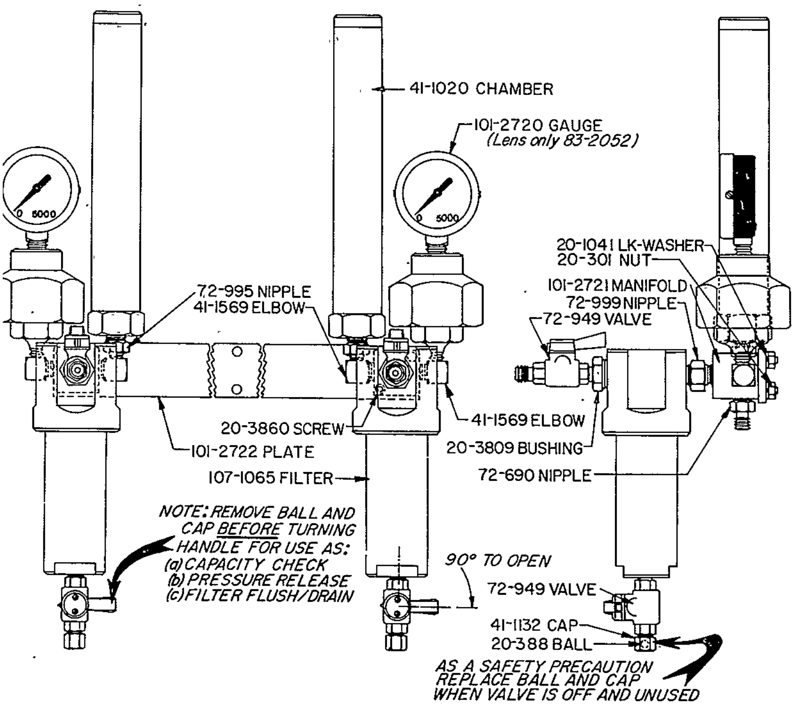


FIGURE 15. HOT HYDRAULIC FLUID MANIFOLD ASSEMBLY

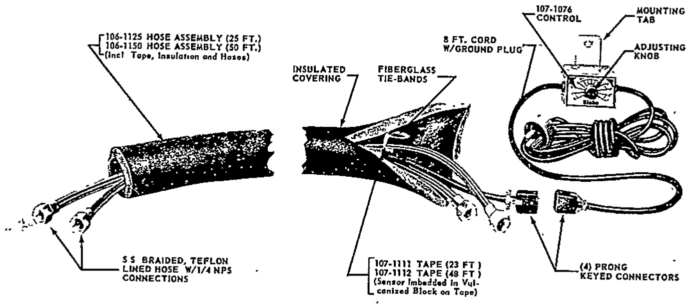


FIGURE 16. HOT HYDRAULIC OUTPUT HOSE ASSEMBLY (HEATED)

5. Foam Spray Gun. The 43P foam spray gun shown in Figure 17 provides the means by which the metered foam components are mixed and atomized. The gun is an airless type, utilizing high fluid pressures and fluid impingement in the mixing chamber to create the turbulence required for intimate mixing of the resins. The mixed components flow through a sapphire orfice and a tungsten carbide nozzle where the mixture is atomized and directed toward a work piece.

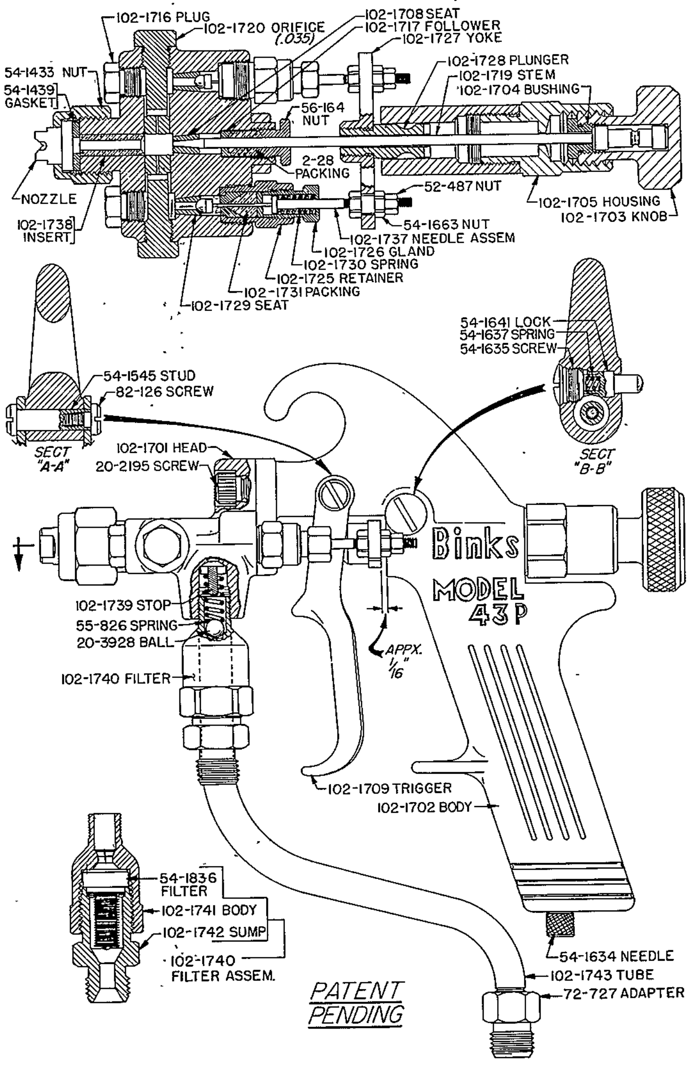


FIGURE 17. MODEL 102-1700 (43 P) AIRLESS FOAM GUN

6. Solvent Flush System. At the completion of spraying operations, after the trigger of the gun has been released the resineous materials must immediately be removed from the mixing chamber and exit openings of the gun. A drawing of the pump solvent flush unit is shown in Figure 18.

The flush system uses an air motor to power the pump shown in Figure 18 which in turn supplies high pressure solvent to the spray gun through a hose connected to the center inlet port of the spray gun. Counter clockwise rotation of the knurled knobs at the rear of the gun opens the valve and allows flow of the high pressure solvent through the mixing chamber and nozzles, thereby removing the resineous materials and leaving the equipment clean and ready for the next spraying operation.

Binks MODELS 103-1032, 5 GALLON & 103-1034, 55 GALLON

25 to 1 Ratio Solvent Flush Pump Units

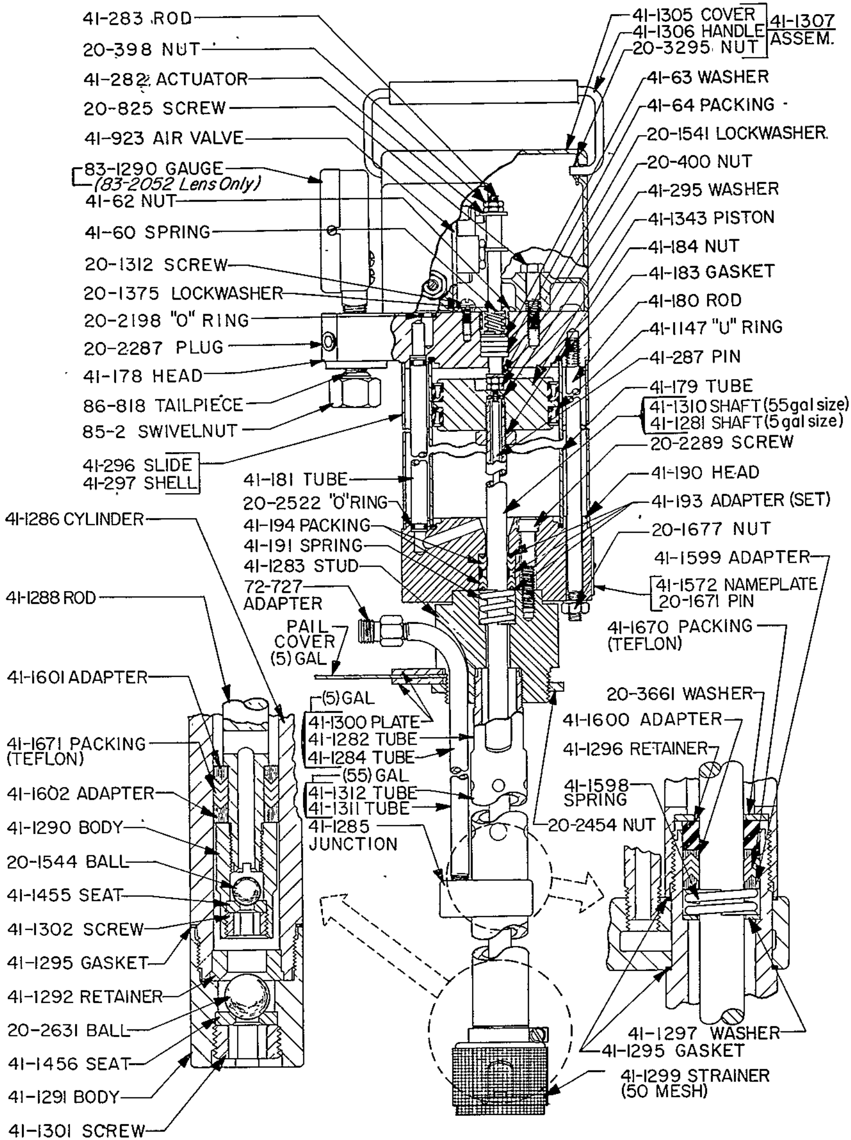


FIGURE 18. MODEL 103-1032 SOLVENT FLUSH PUMP

E. OPERATING PROCEDURES AND CHARACTERISTICS OF THE HOT HYDRAULIC UNIT

Prior to initiation of spraying operations, certain set procedures must be followed in order to be reasonably certain that high quality foam will be applied to the workpiece during foam applications, and in order to have a properly functioning formulator for subsequent foam application, a shut-down procedure must be followed.

1. Pre-Spraying Operations. Prior to initiation of spraying operations, certain precautions and operations must be performed to insure that the formulator will function properly and that high quality foam will result after the two components are mixed.

1. Material Requirements. Although the foam components are produced to the manufacturer's specifications, certain properties of the components should be verified prior to use. These are viscosity, specific gravity, water content, freon content, amine equivalent and hydroxyl numbers.

In addition to the above basic properties of the components, the quality of the foam system should be checked by performing a Test Pour. The properties of the system that can be determined from this are:

a. Cream Time - The time from initiation of mixing until the system begins to react.

b. Rise Time - The time from initiation of mixing until not less than 90 percent of the foaming action has occurred.

c. Tack Free Time - The length of time from the initiation of mixing until the foam ceases to stick to a clean finger of a polyethylene glove.

d. Core Density - The density of the foam after all external skins have been removed.

The materials required to perform the test are an air powered agitator motor with a 12 inch (30.24 cm) stainless steel shaft and a 2 inch (5.04 cm) diameter blade with the vanes adjusted to propel the material in a downward direction, 6 ounce (177.6 cm^3) paper cups (unwaxed), a stop watch, and newspaper.

The test is performed as follows:

a. With both components at $77 \pm 2^{o}F$, weigh 50 grams of component A and then 50 grams of component B into a 6 ounce paper cup.

b. Place under the agitator and simultaneously start the stop watch and agitator.

c. Mix for 3 seconds and pour onto the sheet of newspaper.

d. Record the cream time, rise time, and tack free time. Determine the core density of the foam after 30 minutes.

The values determined can be compared with known values for the foam system in question and an indication of quality obtained. The values for the two foam systems in use by S&E-ME are listed in Table III.

2. Formulator Fill Procedure. In order to insure that the Hot Hydraulic foam spray unit will operate properly and with minimum pulsations, it is required that absolutely no air be present in the system and that the system be filled with the foam components. This condition can be attained by observing the following procedure:

TABLE III MATERIAL SPECIFICATIONS FOR FOAM SYSTEMS IN USE BY S&E-ME

	BX-250A	385-D
Component "A"		
Amine Equivalent	134 ± 3 sec	131 - 13 sec
Viscosity @ 73°F	300 ± 120 @ 73°F	200 - 300 @ 77°F
Specific Gravity @ 73°F	1.235 ± 0.006 @ 73°F	1.23 - 1.25 @ 77°F
Component "B"		
Viscosity @ 73°F	435 ± 150 @ 73°F	450 - 650 @ 77°F
Specific Gravity	1.208 ± 0.012 @ 73°F	1.16 - 1.19 @ 77°F
H_2O Content	0.1 max	0.20 max
Freon Content	28 ± 2	27.7±2
Hydroxyl No.	505 ± 20	---
Refractive Index	---	1.4750-1.4770
Test Pour	100 g @ 73°F	100 g @ 75°F
Cream Time	5 - 8 sec	5 - 7 sec
Rise Time	20 sec 25 max	20 - 30 sec
Tack Free Time	25 sec	17 - 23 sec
Core Density	$2.0 {+0.2 \atop -0.3}$ lb/ft^3	1.75 - 1.90 lb/ft^3

With the pressure pots containing high quality foam components and all valves in the closed position:

a. Remove impingement orifices (102-1720), plugs (102-1716), nozzle, sapphire orifice, and teflon insert (102-1738) from the spray gun if gun has been assembled.

b. Open outlet valves of pressure pots and inlet valves to resin heaters.

c. Open drain valves at bottom of output surge chamber and bleed system until a solid, clear stream of material issues from the valves. Close valves.

d. Pull trigger on spray gun and hold open until a solid clear stream of material issues from each side of the gun. Release trigger and flush gun.

e. Open drain valves at bottom of output surge chamber and verify that a solid stream of clear material issues from the valves. Close valves.

f. Adjust formulator air pressure to 30-35 psi and open valve to air motor of formulator.

g. Pull trigger on spray gun and hold open to verify that a solid stream of clear material issues from each side of the gun.

The spray unit is now filled with material and is ready for use.

3. Ratio Check. The "Hot Hydraulic" system pumps materials in a fixed ratio, however, equipment malfunction (i.e. sticky or leaky liquid end valves) can destroy the proper mix ratio resulting in sub-standard foam. The ratio of the foam components should be verified before foam is applied to any surface. This is accomplished in the following manner:

With the formulator filled with material, the orifices, plugs, and nozzle removed from the spray gun as in Step 1 of the Formulator Fill Procedure, the input air pressure set at 30-35 psi, and the valve to the formulator air motor open; pull trigger on spray gun and hold open until a solid stream of clear material issues from each side of the gun. Hold two tared containers and insert into the streams of material. Care must be taken that the cups enter the material stream at the same time. Collect approximately 100 gm of each component and then release spray gun trigger. Remove cups from vicinity of spray gun and flush gun. The cups should be weighed as rapidly as possible, the cup containing the "B" component first to minimize the error introduced by freon volatilization. The ratio by weight of the components is calculated and compared with the ratio of the component's specific gravity at the temperature at which the samples were collected. A variation greater than $\pm$ 0.003 is an indication than an equipment malfunction exists which must be corrected so that the required ratio can be obtained.

6. Foam Density Check. Prior to application of foam to a workpiece, when the density of a foam specimen is compared to the nominal density of the foam system, an indication is obtained as to whether or not proper density foam is being produced.

One method of rapidly determining foam density is to spray a quart cup full of foam. The top of the foam is cut level with the top of the cup, the cup stripped from the foam (to verify that the cup was full), the foam weighed and the density read from the curve shown in Figure19.. This method produces a reasonably accurate density determination and gives an indication as to whether or not the proper density foam will be produced when sprayed on a surface.

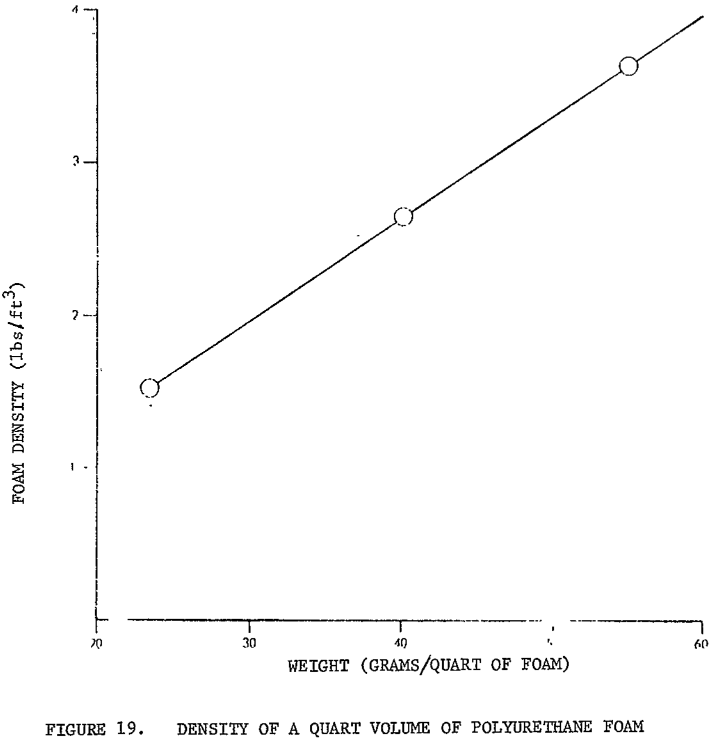


FIGURE 19. DENSITY OF A QUART VOLUME OF POLYURETHANE FOAM

5. Nozzle Selection. The parameter which most greatly effects the output rate of foam from the spray unit (assuming that the unit is being operated at a constant input pressure) is the nozzle size. It was shown in Table II that a wide variety of nozzle sizes are available for use with the 43P spray gun. The foam output rate for several of these nozzles has been determined and the results are tabulated in Table IV.

TABLE IV. FOAM OUTPUT RATE FOR SEVERAL DIFFERENT SIZE SPRAY NOZZLES

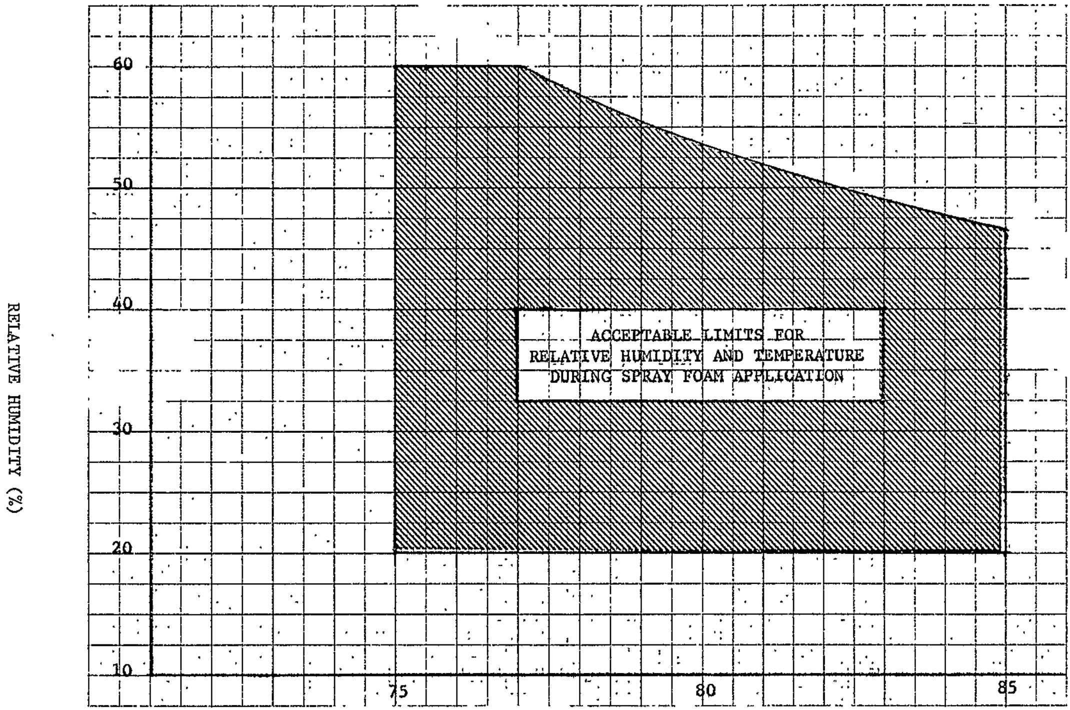
Nozzle Size (in.)	Sapphire Orifice (in.)	Output Pressure (psi)	Output (#/min.)
0.015	0.020	2600	3.0
0.018	0.020	2500	3.0
0.021	0.020	2500	3.4
0.026	0.026	2300	4.8
0.031	0.026	2300	5.4
0.036	0.026	2200	5.7
0.036	0.035 *	2250 **	7.8
0.036	0.052 *	2250 **	8.6

* Stainless steel orifice used because sapphire orifice not available in this size.

** Input pressure raised from 120 psi to a point when atomization occurs.

The above output determinations were made using a spray unit equipped with # 106-1106 liquid ends, a spray gun fitted with "A" - 0.035" and "B" - 0.046" impingement orifices, 120 psi input air except as noted, CPR-385 foam with "A" component at 110°F and "B" component at 80°F.

6. Temperature-Humidity. It has been found that under certain atmospheric conditions (high temperature and high humidity) the foam, as it rises and cures, tears itself away from the surface of the workpiece resulting in little or no adherence of the foam to the surface. The mechanism of this type failure has not yet been entirely established; however, the situation can be eliminated by conducting the spray operations at lower temperatures and humidity conditions. The accepted temperature-humidity values for spray foam operations are shown in Figure 20.



TEMPERATURE (°F)

FIGURE 20. TEMPERATURE-HUMIDITY REQUIREMENTS FOR SPRAY FOAM OPERATIONS

SECTION IV. APPLICATION OF POLYURETHANE SPRAY FOAM INSULATION

There are two basic methods for application of spray foam to a surface: hand application and mechanized application. When spraying on a smoothly contoured body, a mechanized method of application will produce foam having a minimum amount of variation from one given area to another given area. Necessity demands at times that some areas be sprayed by hand, as when an irregular surface is encountered and mechanized methods produce an undesirable foam. Items with which MSFC/S&E-ME is concerned with insulating are, for the most part, confined to cylindrically shaped tanks which lend themselves very nicely to the mechanized application methods discussed herein. Foam is applied to the tank, which is mounted on a movable platform as it rotates in front of an oscillating (up and down) spray gun. After the foam has cured sufficiently the exterior surface is machined to the required configuration and the machined surface sealed with a coating material.

A. SURFACE PREPARATION OF WORKPIECE

Before a surface can be spray foamed, it is mandatory that the surface be thoroughly cleaned and primed if adhesion of the foam to the surface is required. The surface can be cleaned by numerous methods either chemical or mechanical in nature. The most reliable has been a chemical method referred to as the Modified British Etch.

1. Cleaning of Surface. A dirty aluminum article that is to be subsequently spray foamed is prepared for cleaning by wiping the surface with clean gauze or cheesecloth moistened with MEK or acetone to remove foreign matter. Stains or heavy oxides are then removed by sanding with 200 grit wet or dry sandpaper followed by another solvent wipe.

a. Preparation and Application of Etchant. Preparation of these solutions demands the exercise of rigid safety precautions and all contact between the solution and the body must be avoided. Work should be performed in a well ventilated area or use air supply masks when preparing or applying the acid solutions.

The etching solutions are prepared in plastic containers, using the following properties by volume:

a.	De-ionized water	1476 ml.
b.	Add concentrated Nitric Acid (70-78% HNO_3)	378 ml.
c.	Add Ortho-Phosphoric Acid (85-87% H_3PO_4)	1514 ml.
d.	Add Hydrofluosilicic Acid (30% H_2SiF_6)	226 ml.

Stir until mixed thoroughly.

Add Cab-O-Sil, an inert thickening agent, and stir until the desired degree of consistency is obtained.

Apply the acid etching paste with a Nylon brush or a spatula to the cleaned tank surface to a thickness of approximately 1/16 inch. Stir paste frequently while applying. Allow the etchant to react for a maximum of 2 minutes. Then scrape off the paste with a spatula and rinse clean with de-ionized water, until the pH value of the effluent is between 6 and 8.

b. Preparation and Application of the De-oxidizer. The de-oxidizing solutions shall be prepared in plastic containers to the following properties by volume:

a.	De-ionized water	2840 ml.
b.	Add concentrated sulfuric acid (98% H_2SO_4)	588 ml.
c.	Add sodium dichromate	378 grs.
		3428

Stir thoroughly until Sodium Dichromate is completely dissolved and solution mixed.

Add Cab-O-Sil until the desired degree of consistency is obtained. Apply the de-oxidizing paste to the etched area to a thickness of approximately 1/16 inch. Stir paste frequently while applying. Allow the paste to de-oxidize for 5 minutes minimum (45 minutes maximum). Scrape off paste excess with a spatula and rinse thoroughly clean with de-ionized water. Check the pH value of the rinsed surface while wet. The pH level should be between 6 and 8. If it is not, further rinsing should be done until the pH is of the required value.

The cleaned surface should be air dryed and protected with plastic sheeting until priming operations, which should occur within 12 hours after the cleaning is completed.

2. Priming of Surface. Two different primers have been used with the foam systems employed by S&E-ME. They are Dow Corning Primer Z-6020 and Furane Plastics Epocast Primer M.

a. Preparation and Application of Z-6020 Primer

Z-6020 primer is prepared by mixing 0.20 grams of primer with 99.8 grams of absolute ethanol. If a larger amount of the primer is required it shall be prepared in the same proportion by weight. Unused primer should be discarded 8 hours after mixing.

An ordinary paint spray gun is used to apply a thin coat of the mixed primer to the cleaned surface. The sprayed primer should cure for a minimum of one hour at room temperature and should be approximately 0.0005 inch thick.

b. Preparation and Application of Primer M. Prepare the Epocast Primer "M" by thoroughly mixing 1 part of component "C" with 80 parts of component "B" by weight. The unused primer should be discarded 8 hours after mixing. Prepare the thinner for the primer by mixing 30 parts of MEK with 70 parts of Toluene by weight.

For spraying mix 100 parts of resin primer with 180 parts of the thinner by weight.

Wipe the cleaned surfaces of the vehicle to be foamed with MEK prior to applying the primer "M".

Brush or spray the wiped surfaces with the prepared primer "M" to a dried film thickness of 0.0005 inch and allow the primer to cure for a minimum of 4 hours at room temperature (60^{o} to 90^{o}F).

All primed surfaces should be protected with brown paper or plastic sheeting until ready for spray foaming. The foam should be applied to the primed surfaces within 14 days after curing of the primer; however, if this period is exceeded, the primed surfaces can be wiped with MEK and reprimed.

B. SPRAY FOAM OPERATIONS

The method selected for application of polyurethane spray foam to the cylindrically shaped items insulated by R-ME is to spray a circumferential band around the item. The width of the band can be determined by the configuration of the item being insulated. The ends of the band are not connected but are treated as a closeout and later sprayed. The spray guns are re-positioned and a second circumferential band is spray foamed. This procedure is repeated until the entire surface has had foam applied to it. This sequence of events is depicted stepwise in Figure 21.

A bulkhead is insulated in a similar manner. A band is foamed circumferentially around the bulkhead followed by other bands until the bulkhead is completely covered with foam. In actuality, the bulkhead would probably be insulated before the sidewall.

1. <u>Closeouts</u>. The detrimental effect on the mechanical properties of the foam encountered by overlapping foam surfaces can be eliminated by treating the overlap area as a closeout. These overlap areas are encountered at the end of a band of foam when the first sprayed foam surface again rotates in front of the spray gun and also at the edges of a band when the next band is sprayed, and are treated in the following manner.

After a sprayed foam surface has been allowed to cure for a minimum of four hours, the maskants can be removed. The edges of the foam are then trimmed using a phenolic-fiberglass knife, so as not to damage the metal substrate, and sanded, using 200 grit sandpaper, until the foam surfaces are smooth. The sanded surfaces are vacuumed to remove all dust and then coated with a 0.010 to 0.020 - inch thick layer of Narmco 7343/7139 or Adiprene L-100/Moca adhesive as shown in Figure 22 and the adhesive is allowed to cure for a minimum of 12 hours and a maximum of 24 hours. The sprayed foam surfaces adjacent to the closeout are masked to prevent overspray during spraying of the overlap area.

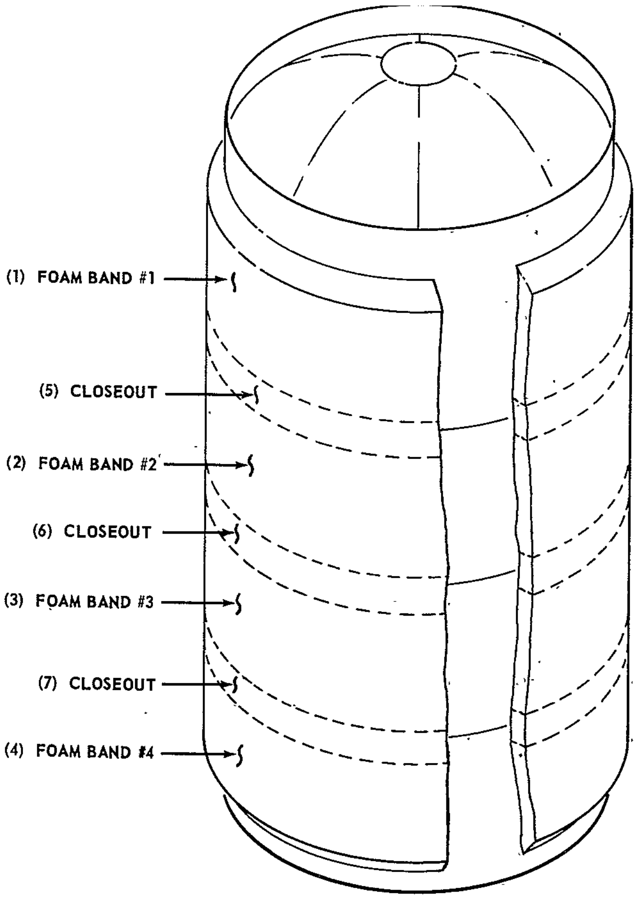


FIGURE 21. SEQUENCE OF OPERATIONS IN SPRAY FOAMING A CYLINDRICAL TANK

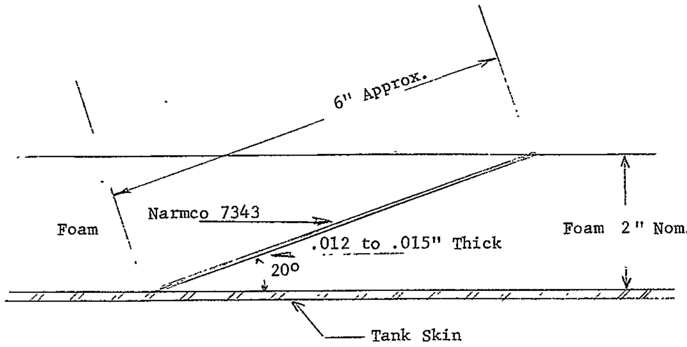


FIGURE 22. LAP-TYPE CLOSE-OUT.

Teflon tape is located at the foam tank skin interface so as to prevent the adhesives from running down onto the tank skin. After the adhesive has cured to the "B" stage, the teflon tape is removed. It is of utmost importance that the adhesive does not run down onto the tank wall as on subsequent cryogenic exposure, a debond will most likely result, requiring a repair.

Following the twelve hour cure, the closeout area is spray foamed. After a four-hour minimum delay the maskants are removed and the overlap is sanded level with the adjacent foam surfaces.

Another type closeout which results in a butt joint is illustrated in Figure 23. The perpendicular edge of the foam is formed either by cutting the foam with a fiberglass knife or by locating a barrier such as an angle to stop the deposition of sprayed foam at the desired place. The edge is sanded and vacuumed as was done with the lap joint.

Teflon tape is located at the foam - tank skin interface, the adhesive is applied to the cut foam surface and after a partial cure has been effected the tape is removed. Foam surfaces adjacent to the closeout were masked and after the adhesive has cured for 12 hours minimum,the joint is completed.

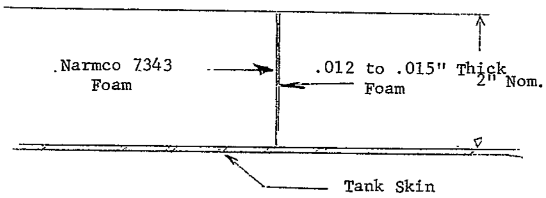


FIGURE 23. BUTT-TYPE CLOSE-OUT.

2. <u>Preparation of Sample Panels.</u> In order to verify the quality of the foam being applied to a surface, "start" and "stop" panels are spray foamed under the same conditions as the article being insulated. The mechanical properties of the foam panel are determined and compared, thereby giving an indication of the quality of the foam applied between the time the panels were sprayed.

An arrangement such as is shown in Figure 24 can be used when spraying on a cylindrical surface. The required number of test panels are affixed to a masked surface with approximately 10 to 12 feet of the surface exposed between the panels. Spray applications are so initiated with the foam falling on the maskant. The surface is rotated so that the "start" panel, the exposed area, and the "stop" panel has been sprayed.

After four hours maximum the vertical edges of the foamed area, are trimmed, prepared for spraying and the "start" and "stop" panels and the foam covered maskant removed. The foam is masked and panels applied to the maskant as shown in Figure 24.

Initiate spraying operations on the maskant and rotate the new "start" panel and the remainder of the belt before the spray gun.

Prior to reaching the left rimmed edge of the foam over area "A" remove P2 and P4 paper with foam and cut the P3 paper to the trimmed left edge of the foam and continue spraying over the surface of the stop test panels and stop at about the middle of the 0.020 aluminum strip. Allow the foam to cure a minimum of 4 hours then cut it with a knife right over the 0.020 aluminum strip. (See Step 3, Figure 24)

Cut the foam with a phenolic fiberglass knife along the edges of the trimmed P3 paper and remove this paper along with the foam, 0.020 aluminum strip and start and stop test panels then cut out the test panels. After a minimum of 24 hours, the panels can be cut up and tested.

As has been described, every time the spray gun is activated, a "start" panel should be made; and every time before the gun is turned off, a "stop" panel should be made.

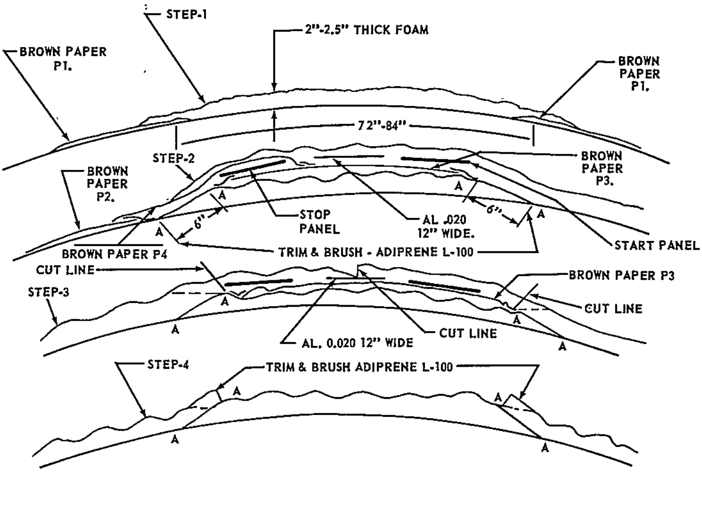


FIGURE 24. SEQUENCE OF EVENTS IN PREPARATION OF FOAM SAMPLE PANELS

SECTION V. EXTERIOR FOAM SURFACE COATING

After completion of the application of the spray foam insulation to the surface of the article being insulated, the surface of the foam is pebbly at best and probably contains a large number of surface discontinuities. If a need or requirement exists for these discontinuities to be removed, the foam surface must be sealed. Also, the surface must be sealed if the insulation is to expand to cryogenic temperatures in order to prevent cryo-pumping.

To date, all test items insulated by R-ME have been sealed with Narmco 7343/7139 or Adiprene L-100. This adhesive apparently provides an effective moisture barrier and seals the polyurethane foam in a satisfactory manner. There are, however, several different methods of application of the coating.

A. SPRAY POLYURETHANE COATING

The easiest method of sealing the foam insulation is to spray a coating of polyurethane adhesive over the as-sprayed surface of the foam. This can be accomplished by changing the normally encountered curing agent for Narmco 7343 or Adiprene L-100 (Narmco 7139 or Moca, and 4-methylene-line-2-chloro aniline) to MDA (Methylene dianiline) and adjusting the concentration of the resin and curing agent so that a one ratio between the two will be required for complete curing.

1. Preparation of Polyurethane Coating. The preparation of the components of the sprayable coating is accomplished by dissolving both the resin and the curing agent in a suitable solvent, in this case ethyl acetate, urethane grade. Then solutions are prepared according to the following directions:

a. Curing agent preparation. The curing agent is prepared by dissolving 62.4 parts by weight of methylene dianiline (MDA) in 853 parts by weight of ethyl acetate, urethane grade, and thoroughly stirring until all of the MDA is dissolved in the ethyl acetate.

b. Resin preparation. The resin portion of the coating is prepared by dissolving 650 parts by weight of Narmco 7343 or Adiprene L-100 in 348 parts by weight ethyl acetate, urethane grade, and stirring until the resin is dissolved in the ethyl acetate. (The resin can be dissolved in toluene, in different proportions, instead of in ethyl acetate. In order to use only one solvent in both sides of the system, ethyl acetate was chosen.)

2. Coating Application Equipment. When the resin and MDA are prepared in the aforementioned proportions, a complete cure of the system will be achieved when they (resin and MDA) are mixed in a ratio of one to one by volume. This can be accomplished by metering them with a Binks Formulator C and spraying the mixture through a 43P spray gun identical to the one used to spray the foam components.

The Formulator C is a "Hot Hydraulic" formulator without any heaters or modifications used on the foam unit. A diagram of this system is shown in Figure 25.

3. Application of Sprayable Polyurethane Coating. The resin and curing agent are prepared as described previously and placed in proper sized pressure pots, the curing agent on the "A" side and the resin on the "B" side and the pots are pressurized with 100-110 psi dry nitrogen. The solvent flush pot is filled with ethyl acetate and the coating sprayed onto the foam in a manner similar to the application of the foam.

The build-up of the coating should be approximately 0.005 inch per pass. A delay of five to ten minutes after application of a coat allows the sprayed material to cure sufficiently for application of another coat. A coating 0.010-0.015 inch thick has been found to perform satisfactorily. After application of the coating, the insulated article should be allowed to cure for seven days at room temperature.

B. FIBERGLASS REINFORCED POLYURETHANE COATING

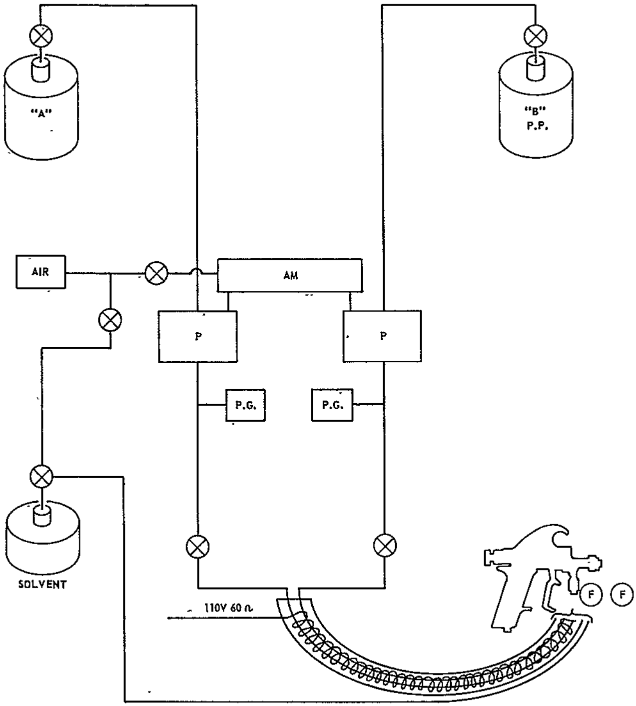
If the surface of the foam has been machined, it is necessary to use a 100% solid adhesive system because of the detrimental effect of solvent on the cell structure of cut foam.

1. Preparation of Adhesive. The same Adiprene L-100/Moca or Narmco 7343/7139 proportions as were used in preparation of closeout edges are used for the outer coatings (100 parts Adiprene by weight to 12.5 parts Moca by weight). The Moca curing agent is melted at $250^{\circ}{}^{+0}_{-10}{}^{\circ}F$ and mixed thoroughly with the Adiprene.

2. Application of Adhesive and Fiberglass. The mixed Adiprene L-100 or Narmco 7343 is applied to the foam surface and spread either by brushing, roller coating or trowelling until a smooth layer of the adhesive, approximately 0.010 to 0.020-inch thick, has been spread over the area to be coated. The adhesive is allowed to partially cure, and while it is still tacky, a layer of #118 glass cloth saturated with the Adiprene is applied to the resin coated surface and all wrinkles and bubbles carefully worked out of the cloth.

The resin-glass layer is allowed to cure for 24 hours after which a second layer of resin saturated cloth is applied to the surface. After the second layer has cured for 24 hours, a final coat of adhesive is applied to the glass covered surface.

This final adhesive layer is allowed to cure for 24 hours after which, if desired, the surface can be painted.



P.P. - PRESSURE POT

P.G. - PRESSURE GAUGE

AM - AIR MOTOR

P - PUMP

FIGURE 25. DIAGRAM OF EQUIPMENT USED FOR APPLICATION OF SPRAYABLE POLYURETHANE COATING

SECTION VI. TEST ITEMS INSULATED WITH POLYURETHANE SPRAY FOAM

Several items have been insulated with polyurethane spray foam in order to verify the application equipment, method of application; and to test the in-place insulation system at cryogenic temperatures. A brief description of each insulation system and the results of testing the system are presented in the following paragraphs:

A. 70-INCH DIAMETER TEST TANK

The first item insulated with polyurethane spray foam by S&E-ME, was a 70-inch diameter test tank. This tank was insulated for the purpose of ascertaining whether or not the Binks "Hot Hydraulic" foam unit could be used to successfully apply cryogenic insulation and to check the quality of the insulation.

1. Method of Foam Application. The foam system employed in this project was CPR 369-3, the system then under consideration for use on the S-II stage. The application equipment used to apply the CPR 369-3 foam was the Modified Hot Hydraulic unit, shown in Figure 7, equipped with a 106-1106 liquid end on the "A" side and a 106-1105 liquid end on the "B" side.

The tank was etched and de-oxidized using the Modified British etch system and was primed with a silane type primer.

The primed tank was mounted on an appropriately sized turntable located on the floor of the Vertical Assembly Tower and the spray gun secured to a tool post on the movable platform. The tool post was fixed to the extent that the gun did not oscillate; it could, however, be raised or lowered approximately 3 ft. on the post.

Rotation of the tank was initiated and the spray gun was actuated with the gun pointed directly at the tank. Foam was applied to the tank until a band of foam slightly greater than 2 inches thickness was achieved. The gun was de-actuated, rotated 90° on the tool post, flushed and returned to its original direction for spraying the next band. The elevator was raised to enable the next band to be sprayed (or, if possible, the gun was raised on the tool post) and the next band was foamed. This procedure was repeated until the entire tank had been spray foamed in an upward direction from the bottom to the top.

2. Outer Covering. After the prescribed cure cycle for the foam had elapsed, the surface of the foam was machined and EC 2216 was applied as a spray until an outer covering thickness of approximately 0.015-inch was obtained.

3. Test Results. The tank was subsequently moved to the liquid hydrogen test facility, and a liquid hydrogen fill and drain cycle was conducted. When warm-up of the tank to ambient conditions had been achieved it was noted that foam failures were noted in several places on the tank. These failures were traced to small amounts of solvent remaining in the mix chamber of the gun after flushing so that when the gun was later sprayed directly at the tank the small amount of solvent would be trapped in the foam causing sub-standard foam in that area. This condition was rectified by actuating the gun on a piece of paper, catching the solvent filled foam on the paper, then shifting the spray foam on to the tank surface.

The tank was removed from the test cell and the sub-standard foam areas repaired. Prior to re-testing the CPR 369-3 foam system was dropped from consideration for use on the S-II stage; thus no additional fill and drain cycles were performed on this tank.

The test did indicate, however, that the "Hot Hydraulic" formulator could be used satisfactorily for foam application.

B. 30-INCH CUBICAL DEWAR

In order to evaluate the effect of nuclear radiation on the candidate insulation systems for the Nuclear Ground Test Module, a 30-inch cube shaped dewar, designated as Cube "A", was fabricated and insulated with cork and spray foam. A second cube designated as Cube "B" was later fabricated and insulated with only polyurethane spray foam.

1. Insulation of Cube "A". Cube "A" was fabricated by S&E-ME-D and delivered to S&E-ASTN-M where two side faces of the cube were insulated with cork. The dewar was then returned to S&E-ME-M where the remaining two sides and the bottom were insulated with polyurethane spray foam.

a. Method of Foam Application. Upjohn CPR 368-2 spray foam was applied to the 30 inch cube tank using the modified Hot Hydraulic spray foam equipment. No facilities existed at that time for controlling the humidity and temperature of the spray area and no attempt to control these parameters was made.

The two cube sides which were already insulated with cork were masked with polyethylene film. The remaining portions of the tank, other than the bottom, were individually masked with polyethylene film so that one side could be foamed without removing the maskant from the area not yet insulated.

The cube was positioned on an unfoamed side and the spray foam was applied to the bottom of the cube in three layers of approximately one-inch thickness per layer. The foam spray was applied to the tank using a back and forth horizontal motion. The tank was then placed in an upright position, the maskant removed from one of the uninsulated cube faces, and foam applied to that face in a manner similar to that employed in insulation of the bottom. The remaining side and the helium shroud area at the top of the tank were insulated in the same manner.

b. Outer Covering. The surface of the foam was machined, using a hand router until the desired thickness (2 inches, + ¼, -0) was obtained. The machined foam surface was then covered with two layers of Narmco 7343/7139 impregnated, No. 116 glass cloth. After the 7343-glass cloth covering had partially cured (24 hours after application of second layer) the tank was painted with two coats of S-13 white epoxy paint and shipped to General Dynamics/Ft. Worth for testing. A photograph of the insulated dewar is shown in Figure 26.

c. Test Results. Safety regulations of General Dynamics/Ft. Worth required that the cube be subjected to one LN_2 fill and drain cycle and two LH_2 fill and drain cycles prior to exposure of the cube to the reactor. Each cycle consisted of cool-down of the dewar, filling of the dewar, draining the dewar, and warm-up of the dewar. This sequence of events required 6½ to 8 hours to complete. At the completion of the above three cycles, there was no observable degradation of the insulation. The cube was moved into the reactor area, filled with LH_2, and the reactor brought up to power. After approximately 8 hours exposure of the cube to the nuclear radiation environment, two distinct explosions were heard and testing of the dewar was stopped at that point.

Inspection of the cube revealed that the cork had exploded and burned. The damaged cube is shown in Figure 27. Further inspection of the cube revealed that the foam still adhered to the surface of the cube and did not indicate that the foam was involved in the initiation of the explosion.

2. Insulation of Cube "B". An evaluation of the conditions surrounding the failure of the Cube "A" insulation indicated that the foam would probably be a usable insulation system. The decision was made at that time to fabricate and test a second, similarly configured dewar. The CPR 385-2 foam system was used for the insulation.

a. Method of Foam Application. The shroud and bottom of the tank were spray foamed using the modified Hot Hydraulic spray foam unit shown in Figure 1 and a hand held gun application technique. Instead of applying the full thickness of the foam in one pass, the desired thickness was built up in 3 passes of approximately 1-inch thickness each. The horizontal, back and forth motion was again employed for the foam application.

The edges of the applied foam that would come in contact with the sidewall insulation when it was applied were then smoothly cut with a knife to eliminate the laps and folds encountered at the sprayed edges.

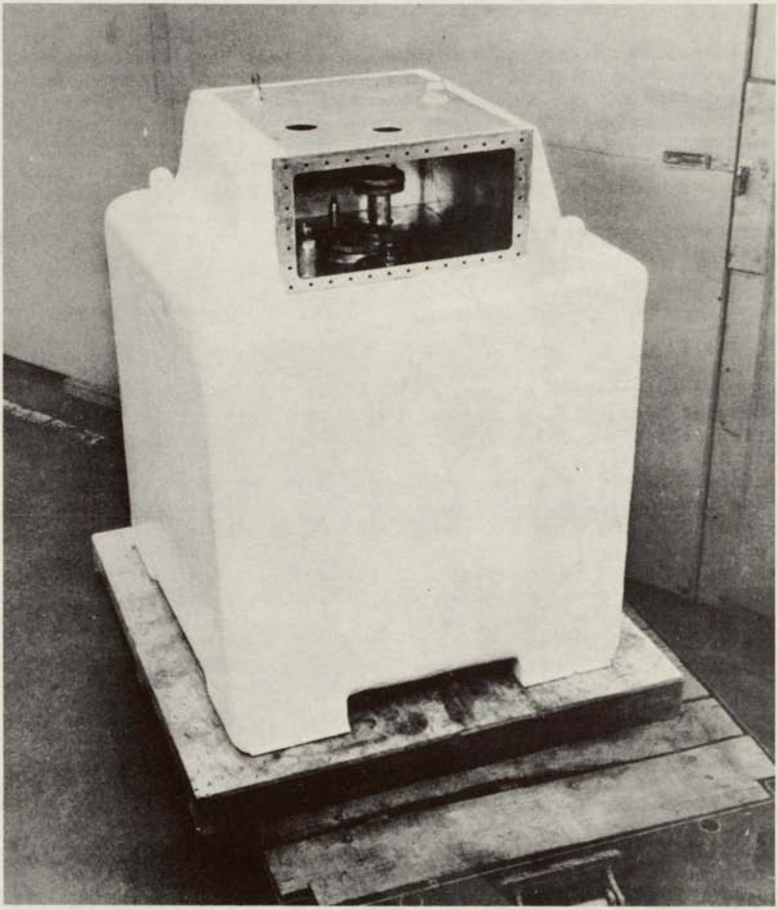


FIGURE 26. CUBE "A" PRIOR TO TESTING

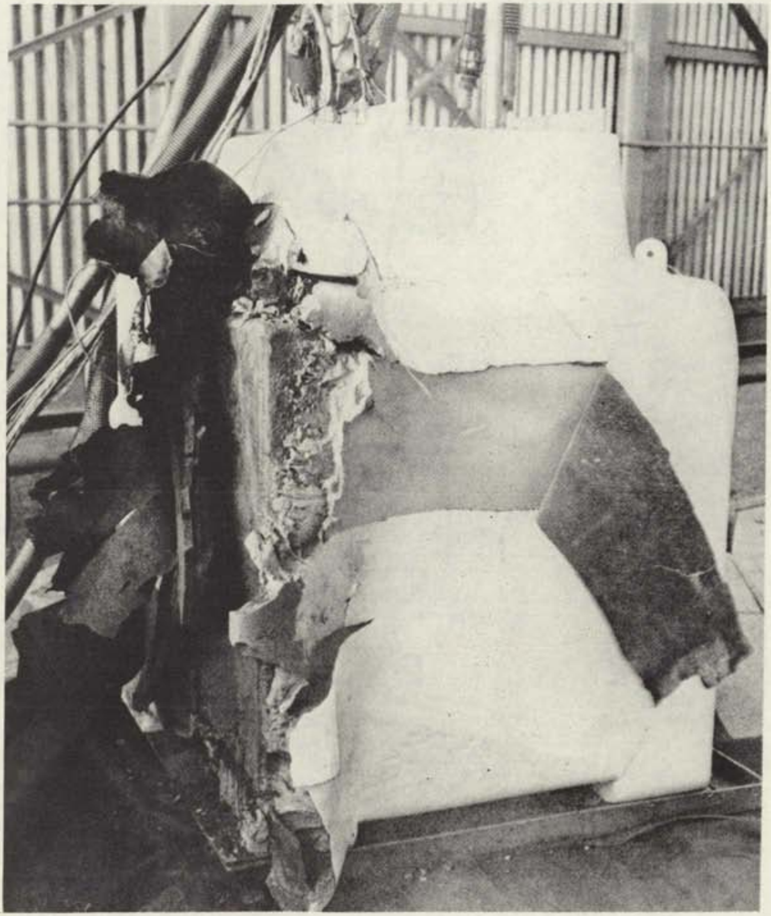


FIGURE 27. CUBE "A" AFTER TESTING

During the trimming of the top and bottom edges it was found that there was no adhesion of the foam to the weld zones. The bad foam was cut away and all weld zones coated with Narmco 7343 at the same time the edges were coated. The resin was allowed to cure for 12 hours at which time the side of the cube was spray foamed in a manner similar to that used to apply the foam to the shroud and bottom of the tank.

b. Outer Covering. The foam surface was machined using a hand router to a thickness of 2 inches, + ¼, -0, and the machined foam surface sealed with 2 layers of Narmco 7343/7139 impregnated No. 116 glass cloth followed by 2 coats of white epoxy paint after the resin had cured.

Cube "B" was then shipped to General Dynamics/Ft. Worth for testing.

c. Test Results. The dewar was subjected to one LN_2 fill and drain cycle and two LH_2 fill and drain cycles prior to radiation exposure. At the completion of the three cycles there was no observable degradation of the insulation.

The tank was moved into the reactor area, filled with LH_2, and the reactor brought up to power. This portion of the test required approximately 36 hours to complete. During this time the liquid level of LH_2 in the dewar was maintained.

After the completion of this phase of the test, the reactor was shut down; the LH_2 drained from the cube; the cube filled with LN_2 and removed from the reactor.

At this point there was still no observable degradation of the insulation.

The cube was removed from the vicinity of the reactor, and an acoustic enclosure placed over it. An acoustic horn was attached to the enclosure and the cube, filled with LN_2 was subjected to six each, one hour exposures to acoustic excitation at a level of 140 DB. The cube was examined at the end of each one hour test for damage and none was observed.

At this point scheduled testing was completed with the insulation having been cryogenically exposed for approximately 120 hours with no observable degradation of the insulation.

Liquid hydrogen fill and drain cycles were run on the dewar after completion of the tests. After the third cycle, two debonds were noted, one on a side and one in the shroud area. Analysis revealed that they were cracks encountered when the foam is punctured prior to sealing such as probe holes used to determine the foam thickness. The debond on the side of the cube is shown in Figure 28.

FIGURE 28. CUBE "B" AFTER TESTING

C. S-IC/S-II TEST CONTAINER

The S-IC/S-II Test Container currently undergoing testing is the first 33 ft. diameter tank insulated with polyurethane spray foam by S&E-ME.

The structure had two different foam systems applied to it; CPR 385-2 on the upper bulkhead and S-IC portion of the cylindrical structure, and BX-250A on the S-II portion of the cylinder.

1. Method of Foam Application. Because of the size of the tank and the use of the two foam systems the application of the foam was divided into three distinct steps. The upper bulkhead was insulated first followed by the S-II sidewall.

a. Application of Foam to Bulkhead. The upper bulkhead was divided into three bands which were insulated individually beginning with the top band followed by the lowermost band, after which the center band was filled in. Three air actuated spray gun oscillators were used to foam the top band and two oscillators were used on the lower two bands. Each band was joined to the one adjacent to it by overlap type joints.

b. Application of Foam to S-IC Cylindrical Section. Upon completion of the bulkhead insulation the forward shirt was installed and CPR 385-2 foam applied to the S-IC sidewall between the S-IC/S-II weld and the S-IC/Y-ring weld. The foam edge at the S-IC/Y-ring weld was prepared for an overlap type joint; and the Y-ring and approximately 18 inches of the forward skirt were foamed again using CPR 385-2 foam. Two air actuated oscillators were used in both cases.

c. Application of foam to S-II Cylindrical Section. The sidewall from the S-IC/S-II weld downward was insulated with BX 250A foam in two steps. The portion from the S-IC/S-II weld to the top of Cylinder 1 was foamed using two air actuated oscillators and the portion from the top of Cylinder 1 to the bottom of the bolting ring was then insulated also using two oscillators.

All sidewall welds were left exposed and prepared for a butt type closeout. The void areas were filled with pre-formed foam blocks and the blocks sealed on the outer surface so that after cryogenic exposure the pre-formed blocks could be removed and welds inspected.

2. Outer Cover. All of the foam surfaces were left in the "as - sprayed" condition and were sealed with three spray coats of MDA cured Adiprene L-100. This resulted in an outer covering of 0.015 to 0.020 inch thickness.

3. Test Results. After the first cryogenic tanking, numerous splits were found between and on top of the stringers in the bolting ring area. In addition two debonded areas were located approximately at the top of Cylinder 1 which could have been caused from cryopumping into a foam block filled weld area where the debonds were probably initiated. The bolting ring failures were probably caused from a combination of voids at the top of the stringers and the use of low purity ethyl acetate in the Adiprene L-100 coating. All defective areas were removed and repairs were made to these area before the second cryogenic cycle.

D. 105 INCH DIAMETER RIFT TANK

A 105 inch diameter tank, designated the Rift Tank (it was originally fabricated for use during the Rift program) was also insulated for use as Government Furnished Equipment in Contract NAS8-18024, "Evaluation of Cryogenic Insulation Materials and Composites for use in Nuclear Radiation Environment."

1. Method of Foam Application. The Hot Hydraulic formulator was used to apply CPR 385-2 foam as the insulation system.

The lower bulkhead was insulated using one air actuated spray gun oscillator and the edge of the foam at the bulkhead/cylinder weld was prepared for overlap type joint using Narmco 7343/7139.

The cylindrical section of the tank was spray foamed using two air actuated spray gun oscillators with the tank rotating at a speed of approximately 35 inches/minute so as to eliminate the requirement for a vertical close-out. The foam edge at the cylinder/upper bulkhead weld was prepared for an overlap type joint using Narmco 7343/7139.

The upper bulkhead was foamed using two air actuated spray gun oscillators. The gun locations and distances were determined empirically and verified experimentally prior to foam application in all cases.

2. Outer Cover. After cure of the foam, its outer surface was machined, using a hand router on the bulkhead and the "Lawn Mower"* on the cylindrical section, to a thickness of 2 inches + ½, -0. The machined surfaces were covered with two layers of Narmco 7343/7139 impregnated No. 116 glass cloth. After curing for 24 hours (minimum) the tank was painted with S-13 white epoxy paint. The tank is shown in Figure 29.

3. Test Results. The tank is awaiting testing at General Dynamics/Ft. Worth.

*Similar to a wood plane.

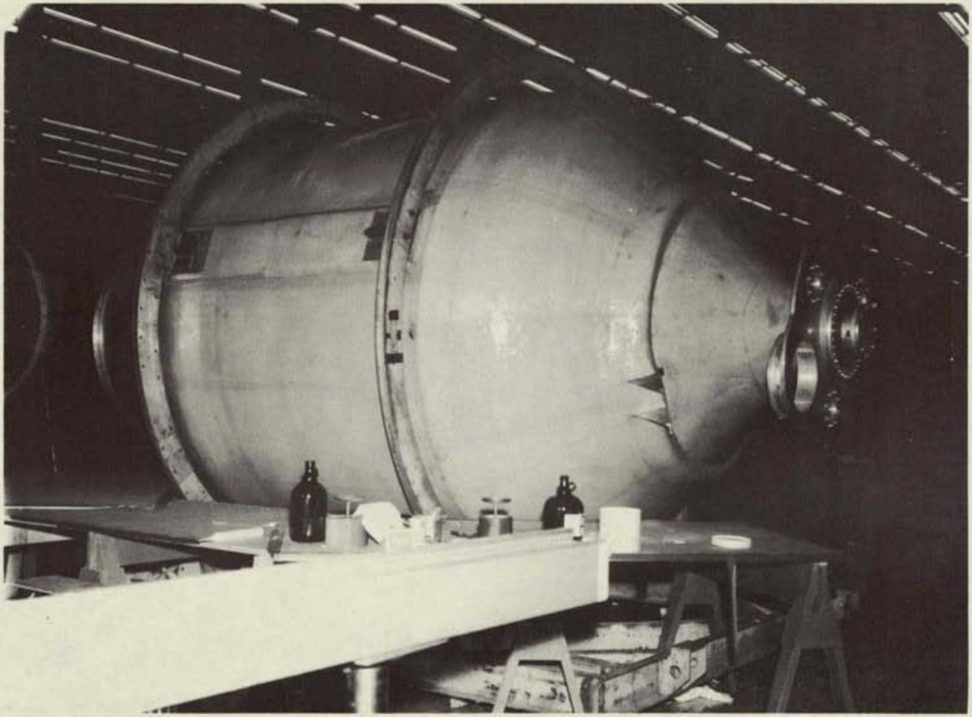


FIGURE 29. 105-INCH DIAMETER RIFT TANK AFTER APPLICATION OF FOAM INSULATION

SECTION VII. CONCLUSIONS

It is concluded that the Manufacturing Engineering Laboratory has developed the capability to satisfactorily apply polyurethane spray foam insulation to cryogenic propellant tanks.

The Binks Manufacturing Company Hot Hydraulic Spray Foam Formulator #105-1027 has been selected for use, and when certain modifications have been made to the basic unit, it has proven to be a dependable piece of foam application equipment.

Foam application methods have been determined, tested on subscale tanks, and then used to successfully insulate a 33-foot diameter liquid hydrogen tank with polyurethane spray foam.

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IN-ME-69-3

APPROVAL

DEVELOPMENT OF METHODS FOR APPLICATION OF POLYURETHANE SPRAY FOAM INSULATION SYSTEMS TO LIQUID HYDROGEN TANKS

By

James M. Carter

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

W. A. Wilson

W. A. WILSON
Chief, Mechanical and Chemical Development Branch

J. P. Orr

J. P. ORR
Chief, Manufacturing Research and Technology Division

M Siebel

MATHIAS P. ~~L.~~ SIEBEL
Director, Manufacturing Engineering Laboratory

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