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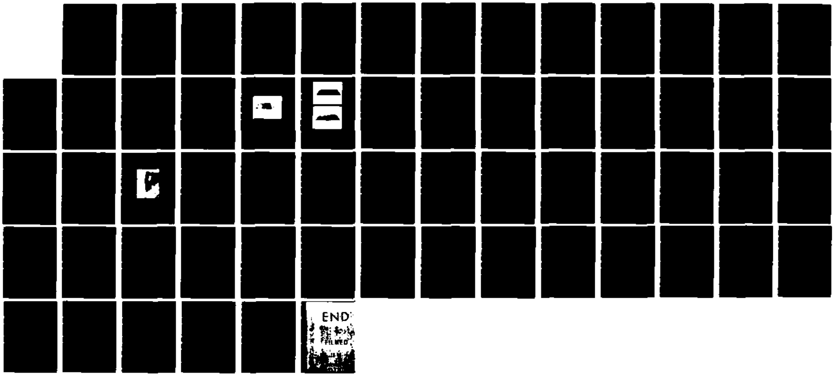

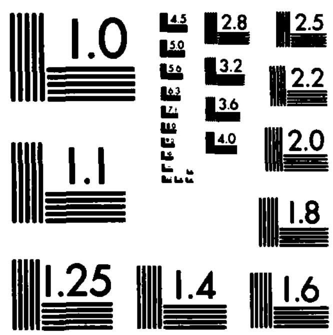
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Johns-Manville
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DEVELOPMENT OF A FIRE-RESISTANT ANTI-SWEAT SUBMARINE HULL INSULATION BASED ON FIBER GLASS MATERIALS

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FINAL REPORT

ON

DEVELOPMENT OF A FIRE-RESISTANT ANTI-SWEAT SUBMARINE HULL INSULATION BASED ON FIBER GLASS MATERIALS

Submitted by:

R. C. Himes
H. Mitchell
S. Baca

MANVILLE SERVICE CORPORATION
RESEARCH AND DEVELOPMENT CENTER
P. O. Box 5108
Denver, Colorado 80217

September 1983

Prepared for:

NAVAL RESEARCH LABORATORY
4555 Overlook Avenue, S. W.
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Development of two candidate submarine hull insulations based on fiber glass materials is described. Properties of the materials, submitted to the Navy in substantial quantity, are presented in detail. The fire-resistant, anti-sweat materials meet or exceed 10 of the 13 specifications for which tests have been performed. Further development is recommended.

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EXECUTIVE SUMMARY

Under Contract No. N00014-82-C-2389, personnel of the Manville Service Corporation's Research and Development Center have executed a program to develop fire-resistant, anti-sweat submarine hull insulation. This was one of five such programs simultaneously sponsored and monitored by the Naval Research Laboratory. The Manville program has resulted in a fiber glass-based product concept which shows substantial promise for the specific application at hand and which could prove to be of value in resolving other insulation-related problems of the Navy. This concept, which involves water repellent, medium-density, low thermal conductivity, fire resistant fiber glass blankets in a composite structure of high integrity, is embodied in two products submitted to the Navy in fulfillment of the contract.

In the products submitted, one-inch-thick flexible blankets (or boards) of roughly three pcf, five-micron fiber glass are sandwiched between woven fiber glass scrims, sewn in place by a quilting technique using fiber glass thread. The core blankets are rendered water repellent by incorporation of certain silicones in the phenolic binder systems employed in their manufacture. The scrimmed structures are designed to be of such tensile strength in the direction normal to the plane that they may be installed by adhesives, rather than by the cumbersome, welded stud systems typically employed with fiber glass insulations.

The products are faced with tough, woven fiber glass structures which incorporate Mylar films. These are employed to provide durable wear surfaces and protected water-vapor barriers. Since integrity of these facings is essential to their functions, freedom from the need to degrade them by impaling on studs for installation is especially attractive.

The products met or exceeded the target property specifications established by the Navy in 10 of the 13 types of tests performed in fulfilling the contract. Requirements for areal density, compression set, dimensional stability, smoke density, oil resistance, tensile strength, flexibility, thermal conductivity, and chemical stability were generously exceeded.

The standards for compression resistance and for water absorption were not met. Reexamination of the appropriateness of those standards is recommended.

Tests of the water-vapor permeability gave results in excess of the standards. Since the facing materials employed have shown excellent (low) permeance, serious questions are raised as to the accuracy of the tests as performed on the thick, flexible composites or as to lot-to-lot variation in the facing materials employed. It seems highly improbable that the handling and adhesive application of the facings could have influenced their permeance.

Seven steps toward refinement of the products and full exploration of the concept on which they are based are recommended.

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

Recent experience has revealed unacceptable fire hazard associated with present day anti-sweat, submarine-hull insulation (Mil-P-15280 H). Generation of dense black smoke, rapid flame propagation, and production of hazardous chemical species have been observed when the insulation is exposed to a representative fire threat. In May of 1982, the U. S. Naval Research Laboratory issued its solicitation N00014-82-R-MO21 for proposals for Phase I of a three-phase program for the Development of a Fire-Resistant Anti-Sweat Submarine Hull Insulation. Manville Service Corporation responded with a proposal to develop such an insulation based on fiber glass blankets, and was subsequently awarded Contract Number N00014-82-C-2389 to proceed with Phase I as proposed. The program was executed in the period 17 September 1982 to 17 July 1983, and resulted in the development of two candidate insulation board products, quantities of which have been submitted to the sponsoring agency for evaluation. This document is the final report on the project.

PROGRAM OBJECTIVE

The stated objective of our work has been to develop a fiberglass-based submarine-hull insulation which will meet the Navy's property specifications and will be amenable to carry-through to successful completion of future Phases II and III.

PROGRAM PLAN

Drawing upon the Manville Corporation's long and broad experience in the development, manufacture, and distribution of special-purpose insulating materials based on polymeric foams, glass fiber, refractory fiber, mineral particulate, etc., we chose to offer the Navy a development program directed toward a fiber-glass-based product which would satisfy the Navy's requirements. For ready reference, we repeat, below, the statement of those requirements (specified for the fire-resistant, anti-sweat hull insulation to be developed), which was presented in the aforementioned Navy solicitation:

> Areal Density - The Contractor shall produce material having an areal density no greater than 0.75 pounds per square foot.
>
> Compression resistance - The Contractor shall produce material having compression resistance at 25 percent deflection of not less than 2.0 pounds per square inch and not greater than 10.0 pounds per square inch.
>
> Water absorption - The Contractor shall produce material having water absorption of no greater than 0.1 pounds per square foot.
>
> Compression set - The Contractor shall produce material having a compression set not greater than 25 percent.
>
> Dimensional change - The Contractor shall produce material having a dimensional change of not greater than 10 percent of its original length.
>
> Fire resistance - The Contractor shall produce material which does not flash over when tested in the Navy 1/4 scale test chamber.
>
> Smoke density - The Contractor shall produce material having a maximum specific optic density of not greater than 250.
>
> Oil resistance - The Contractor shall produce material which does not soften or swell when immersed in oil.
>
> Tensile strength - The Contractor shall produce material having a tensile strength not less than 20 pounds per square inch.

Flexibility - The Contractor shall produce material which is sufficiently flexible to be installed in a 33 foot diameter hull.

Thermal conductivity - The Contractor shall produce material having a thermal conductivity in no case greater than 0.30 Btu inches per hour square foot degree fahrenheit.

Water vapor permeability - The Contractor shall produce material having a water vapor permeability in no case greater than 0.30 perm-in.

Mercury and asbestos free - The Contractor shall produce material which is mercury and asbetos free.

Chemical stability - Material will be chemically stable to 175°F.

The inherently low thermal conductivity of fiber glass blankets at medium density (leading to attractively low ka values) and their inherent chemical inertness make fiber glass structures especially strong candidates. These characteristics provide for great design flexibility in the effort to satisfy the entire array of property requirements without approaching the limits set for key properties such as areal density, fire resistance, smoke density, oil resistance, thermal conductivity, and chemical stability. Conversely, such structures present special problems in the areas of water absorption and water-vapor permeability. The combination of mechanical properties (flexibility, tensile strength, compression resistance, and compression set) required presents an interesting challenge because of the contrary effects upon one property which may derive from parameter variations employed to enhance another.

Our research program directed toward the solution of the multidimensional problem posed was built around the several product parameters amenable to variation by control of the processes for manufacture of the fiber glass blanket. Of principal interest are blanket density and thickness, fiber diameter, fiber lay (dominant orientations), and binder content and type. The binder considerations were of special significance in the subject study. They substantially influence the relationships among the mechanical properties we have mentioned earlier, but probably of greater importance is their role in controlling water absorption. We have invoked proprietary Manville technology in selecting and employing certain silicones to supplement our typical phenolic binders in such a way as to impart valuable resistance to wetting.

The matter of water-vapor permeability has been addressed by use of low-permeance films in the tough facing materials we have employed to provide wear surfaces suitable for shipboard service. In dealing with strength, durability, and installability problems associated with fiber glass insulations in shipboard applications, we have developed an approach, not contemplated at the beginning of the program, to impart good tensile strength in the direction perpendicular to the plane, opening new avenues for installation.

In executing our research program, we have studied properties and parameters of a wide range of existing fiber glass products to assess dependence of the performance-related properties upon the parameters amenable to control in the manufacturing process. With information thus generated we were able to design materials systems to optimize sets of properties of principal concern. A variety of candidate systems (bonded fiber glass blankets) were produced in pilot quantities on full-scale production machinery. The most suitable of these systems were carried through the additional process steps to prepare the samples of candidate materials provided for Navy evaluation in fulfillment of our contract. Two hundred square feet of one product were required. Manville satisfied this requirement and supplied also 100 square feet of a second candidate material to broaden the evaluation base.

The products submitted to the Navy are described in some detail in the following sections of this report, as are the steps which led to their development.

PRODUCTS SUBMITTED TO NAVY BY MANVILLE

Qualitative Description of Products

The development program directed toward satisfaction of the Navy's requirements for a fire-resistant, anti-sweat submarine hull insulation resulted in development of a composite insulation system with several features which may have special utility in areas other than that of direct interest in this program. The products consist of a flexible water-repellent fiber glass board (or blanket), sandwiched between two webs of fiber glass scrim by sew-through quilting, and faced with a tough, paintable wear surface in which is incorporated a water-vapor barrier. These products are intended to be adhesive mountable, obviating need of the welded studs typically used for installation of fiber glass insulations in Naval vessels.

The two products submitted to the Navy for evaluation are designated Product A (200 square feet submitted) and Product B2 (100 square feet submitted). Their compositions are shown in Table 1.

Photographs of the two products are presented in Figures 1 and 2 to augment the above descriptions.

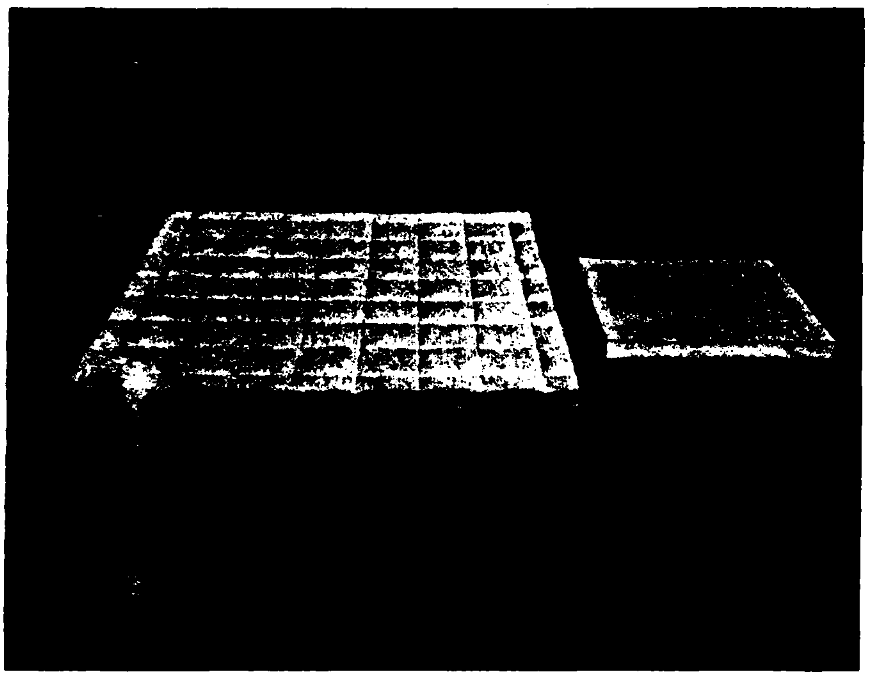
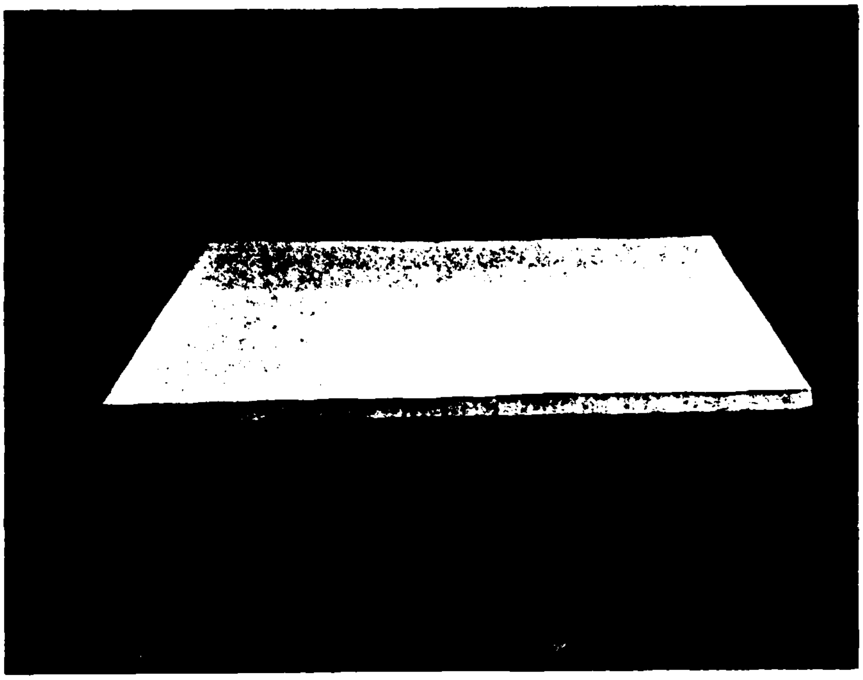


Figure 1. **Pictorial Description of Product A** - Shows both the vapor proof facing side, which would be exposed to the inside submarine environment, and the quilted backside, which would be attached to the hull surface by adhesives.



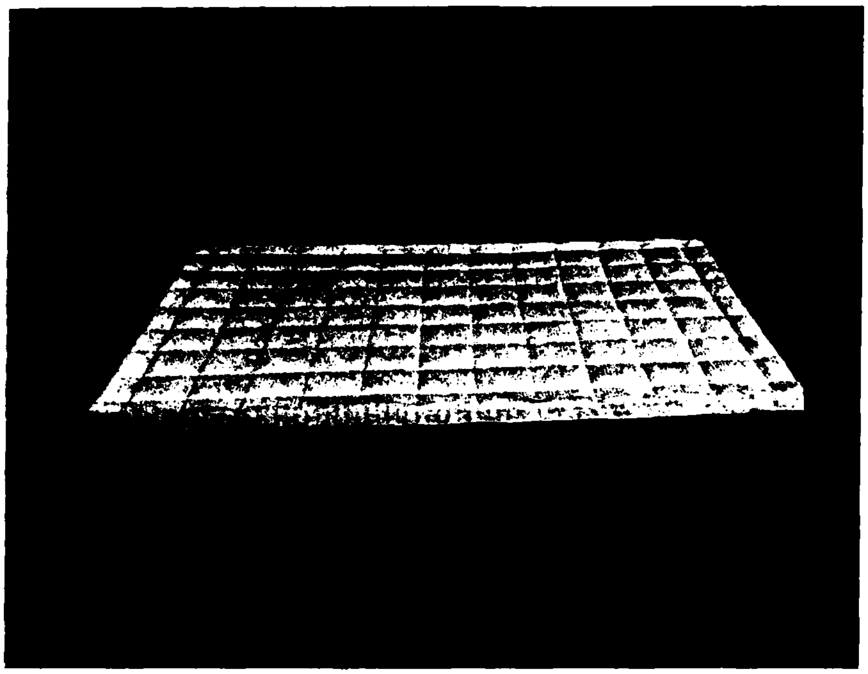


Figure 2. Pictorial Description of Product B2 - Shows the bottom and top surfaces of Product B2 which is similar in appearance to Product A except for the vapor proof facing which is less textured and nearly white in color. As with Product A, the quilted scrim surface is the side which is to be attached to the hull surface by adhesives.

TABLE 1

DESCRIPTION OF PRODUCTS SUBMITTED

Product A

Facing: Claremont 2S14R with 2-mil polyester vapor barrier.

Adhesive: General Latex 3-S716A applied in a dot pattern approximately 12 grams per square foot on the facing.

Scrim: J. P. Stevens 1350/38 Geon 6 x 6 leno weave with PVC finish.

Sewing: E-18 teflon-coated glass thread stitched on a 3-inch x 3-inch pattern.

Fiber Glass Board Product: Three pcr fiber glass with 5 percent phenolic binder and 2 percent added silicone for water repellancy.

Product B2

Facing: Alpha 3267 MAU with 0.5-mil aluminized polyester vapor barrier.

Adhesive: National Starch 72-6800, applied in a dot pattern approximately 12 grams per square foot on the facing.

Scrim: J. P. Stevens 1350/38 Geon 6 x 6 leno weave with PVC finish.

Sewing: E-18 teflon coated glass thread stitched on a 3-inch x 3-inch pattern.

Fiber Glass Board Product: Three pcf fiber glass with 5 percent phenolic binder and 3 percent added reactive silicone for water repellancy.

Test Results and Property Specifications

Fourteen product property specifications are included in the work statement of the contract under which the subject project has been conducted. These were taken as targets for the development effort undertaken. Tests for all but one of the properties identified have been performed. By direction of Navy representatives, the fire testing included in the array of specifications has been reserved for performance in a Navy laboratory. Results of tests performed by Manville are summarized in Table 2. For easy comparison the property specifications are included in the table.

Description of Test Methods and Discussion

Areal Density - The areal density was calculated from the average weight of all sample pieces which were submitted under the contractual agreement. The individual pieces were two feet by three feet for a total of six square feet each. All 34 pieces of Product A were weighed. The average weight for the pieces was 3.20 pounds and the $\sigma = 0.06$ pounds. The average weight for each of the 17 pieces of product B2 was 2.95 pounds and the $\sigma = 0.06$ pounds. A thickness measurement was also performed on each piece using a Gustin Bacon thickness measurement device which consisted of two counterbalanced one square foot parallel plates and a thickness dial gauge. Because the surfaces of a piece of fiber glass board are neither perfectly flat nor perfectly parallel, it is general practice to place a weight on the top plate to gain a good contact with the surfaces. The weight used was 480 grams on the one square foot. The results were a measured average thickness of Product A 1.065 inches with $\sigma = 0.017$ inch. For product B2, the average thickness was 1.086 inches with $\sigma = 0.014$ inch. Without the 480 gram weight, each product would have measured about 1.11 inches with the plate resting on the peak surfaces on the edge of each piece.

Compression Resistance - The compression resistance test was performed on six, 6-inch by 6-inch by thickness, specimens of each product. The load was observed and recorded 60 seconds after a 25 percent deflection was reached using a crosshead speed of one inch per minute.

(ASTM D-1056 recommends crosshead speed on the Instron of 0.5 inch to 2 inches per minute.) The compression resistance in measurements on Product A ranged from 0.75 psi to 1.07 psi for an average of 0.93 psi. Those on Product B2 ranged from 0.85 psi to 1.07 psi and gave an average of 0.94 psi. The compression resistance of fiber glass is normally governed by

TABLE 2

TEST RESULTS AND PROPERTY SPECIFICATIONS

Product A Test Results

Test	Results	Property Specification
Areal Density	0.53 lbs/sq ft	Less than 0.75 pounds per square foot
Compression Resistance	0.9 lbs/sq in	2.0 to 10.0 pounds per square inch
Water Absorption*	Submerged under 25" Hg Vac = 1.2 lbs/sq ft. Submerged at atmospheric pressure under 6" water = 0.08 lbs/sq ft	No greater than 0.1 pounds per square foot
Compression Set	1 percent	No greater than 25 percent
Dimensional Change	0 percent	No greater than 10 percent of its original length
Smoke Density	Flaming mode=61 Non-flaming mode=55	Maximum specific optic density not greater than 250
Oil Resistance	Dimensions remained constant	Does not swell or soften when immersed in oil
Tensile Strength	480 lbs/sq in	Not less than 20 pounds per square inch
Flexibility	Passed	Sufficiently flexible to be installed in a 33 ft diameter hull
Apparent Thermal Conductivity	0.24 Btu in/hr.sq ft ^{o}F	No greater than 0.30 Btu. in/hr.sq ft ^{o}F

Product A - Continued

Test	Results	Property Specification
Water Vapor Permeability	0.9 perm in (Desiccant method) 0.6 perm in (Water method)	No greater than 0.30 perm in.
Mercury and Asbestos Free	No mercury or asbestos used in mfg. of product	Certify material is mercury and asbestos free
Chemical Stability	0.06 percent weight loss	Chemically stable to 175°F weight loss not greater than 1 percent of original weight

Product B2 Test Results

Areal Density	0.49 lbs/sq ft	Less than 0.75 pounds per square foot
Compression Resistance	0.9 lbs/sq in	2.0 to 10.0 pounds per square inch
Water Absorption*	Under 25" Hg Vac = 0.8 lbs/sq ft. Under 6" water = 0.05 lbs/sq ft.	No greater than 0.1 pounds per square foot
Compression Set	0 percent	No greater than 25 percent
Dimensional Change	0.1 percent	No greater than 10 percent of its original length
Smoke Density	Flaming mode = 17 Non-flaming mode = 14	Maximum specific optic density not greater than 250
Oil Resistance	Dimensions remained constant	Does not swell or soften when immersed in oil
Tensile Strength	350 lbs/sq in	Not less than 20 pounds per square inch

Product B2 continued

Test	Results	Property Specification
Flexibility	Passed	Sufficiently flexible to be installed in a 33 ft diameter hull
Apparent Thermal Conductivity	0.23 Btu in/ hr.sq ft °F	No greater than 0.30 Btu. in/hr.sq ft °F
Water Vapor Permeability	0.5 perm in	No greater than 0.30 perm in.
Mercury and Asbestos Free	No mercury or asbestos used in mfg. of product	Certify material is mercury and asbestos free
Chemical Stability	0.12 percent weight loss	Chemically stable to 175°F weight loss not greater than 1 percent of original weight

*Water-absorption data presented here are calculated from experimental measurements in accordance with our interpretation of the wording of Section 4.6.6 of MIL-P-15280H. Thus, "skinless surface" of a 4 x 4 x 1 inch sample with facing material on one 4 x 4 inch face is calculated as 32 square inches (0.22 square feet). If one calculates on basis of "area in the plane" of the sample, the area is 16 square inches and the water-absorption values are twice those in the Table.

ASTM C-165, "Standard Recommended Practice for Measuring Compressive Properties of Thermal Insulations", in which the crosshead speed recommended is 0.05 inches per minute for a one-inch-thick sample. The data reported in the monthly letter reports were based on this crosshead speed and a one-minute relaxation time. The lower crosshead speed was also run on the final product and the results are lower than the D 1056 value. ASTM C-165 states in one of its footnotes, "The speed of crosshead travel can have considerable effect on the compressive resistance value. In general, higher crosshead speeds usually result in higher compressive resistance values." Data from Product A using the 0.05 inch per minute crosshead speed and a two-pound preload ranged from 0.61 to 0.97 and gave an average of 0.75 psi. For the B2 product at the same crosshead speed, data ranged from 0.74 psi to 0.86 psi and gave an average of 0.78 psi resistance after the one minute rest period. As anticipated, the lower crosshead speed did result in lower compression resistance data.

It will be noted that even the D 1056 test results are well below the specified compression resistance value. It is suggested elsewhere in this report a lower value such as we have reported here may prove quite satisfactory for a composite, fiber glass structure of the type with which we are dealing and in the service environment intended.

Water Absorption - Water absorption results are presented for two different tests. The test specificed in Paragraph 4.6.6 of Mil-P-15280H, and in the work statement, seems quite apropriate to a closed-cell foam, for which it is intended. However, that test seems inappropriate for a fibrous or open-cell material in the intended application. The second test, which involves no vacuum exposure, appears more appropriate in the present case.

Water absorption tests and results are discussed in greater detail in a subsequent section of this report, entitled, "Studies on Silicone Additives to Binder Systems".

Compression Set - The samples were compressed 25 percent between parallel plates for 22 hours at room temperature as specified by ASTM D1667-76. The samples were then released from the clamping device and allowed to rest for 24 hours at room temperature. The percentage compression was then calculated from the equation in Paragraph 25.1 of D1667-76. Because of the quilt pattern and the relatively low compression resistance of the fiber glass, we chose to use a sample size of 6-inches by 6-inches. The thickness measurement was made on a Gustin Bacon thickness measurement device to the nearest .001 inch using a preloading of 2.4 ounces. It is standard practice to preload a 3 pcf fiber glass board to gain solid contact with its surfaces.

A second, more stressful test was performed on a second set of samples from Products A and B2. Sample sets of 3 were measured for thickness, compressed 25 percent and held 22 hours at 155°F and 95 percent RH then released and the thickness measured after 45 minutes and again after 24 hours. Product A showed a compression set of 14 percent after 45 minutes and 10 percent after 24 hours. The results for Product B2 under the high temperature and humidity conditions were a compression set of 17 percent after 45 minutes and 13 percent after 24 hours.

Dimensional Change - The dimensional change test was measured in accordance with Paragraph 4.6.8 of Mil-P-15280H. Two three-inch by twelve-inch pieces of each product were cut. Bench marks were placed on the face of each product approximately 10 inches apart. The actual spacing was measured by a Vernier Caliper scale to the nearest .01 inch. Also, on the reverse scrim side of each piece, pins were placed in the sample ten inches apart and measured with a Vernier caliper scale. The pieces were placed in an oven at 200°F for seven days and then remeasured for any dimension change. The only physical change observed was a change in Claremont facing color, to a light brown from its orginal cream color.

Smoke Density - The smoke density was measured in strict accordance with ASTM E-662-79 in both the flaming and nonflaming modes. One noteworthy observation was that the Claremont facing did flame for 30 seconds after exposure while the Alpha facing did not under the flaming exposure test. The average smoke density and standard deviation of each of the three, three-inch by three-inch by one-inch samples tested were:

Product A: $D_m = 55.0$. $\sigma = 0.87$ (nonflaming), $D_m = 61.3$, $\sigma = 0.92$ (flaming)

Product B2: $D_m = 14.5$, $\sigma = 0.46$ (nonflaming), $D_m = 17.3$, $\sigma = 1.55$ (flaming).

Oil Resistance - The oil resistance of each product was determined in accordance with paragraph 4.6.9 of Mil-P-15280H. The exposure of ASTM No. 3 oil did not cause any dimensional change observed with instrumentation accurate to + 0.01 inch. Each one-inch by one-inch by two-inch sample did absorb oil in its open cell structure. The sample weight of 3.2 grams before increased to 32 grams after absorption of the oil for both products. A comparison was made to a standard 3 pcf marine board to detect any influence the silicone binder had on oil absorption. The submitted products exhibited the same oil abosorption as a standard marine board, despite the great disparity in water absorption.

Tensile Strength - The tensile strength measurement of the fiber glass composite deviated in the specimen size from the requirements of D412-80, "Standard Test Method for Rubber Properties in Tension". The most suitable specimen was six-inches by three-inches by the thickness of each product. A notch was cut into each side of the specimen to leave a one-inch wide test area. The crosshead speed of the Instron was set at 12inches per minute, rather than 20-inches per minute as called for in the D-412 test method for rubber. A recorder noted the force applied up to rupture. Paragraph 4.6.11.2 of Mil-P-15280H states all values shall be reported. These are recorded in Table 3.

Flexibility - Both products were able to conform to the 24 foot diameter curvature of the "sub hull test fixture" devised for the subject program. This fixture, devised to impose a conformability test somewhat more severe than the actual specified requirement, is shown in Figure 3. Both product samples passed the flexibility test in Mil-I-22023C. In this test, sample pieces 12 by 18 inches by product thickness are bent on their 12-inch dimension over a 1-inch diameter mandril long enough to extend at least one inch beyond the ends of the test specimen. They are bent through an arc of 90^{o}F, held in the flexed position for five minutes, and released. Subsequently, specimens are examined for rupture or visible cracking. The matter of flexibility, or bending resistance, is treated in greater detail in a subsequent section of this report entitled, "Factors Which Control Fiber Glass Product Performance Characteristics".

Apparent Thermal Conductivity - The apparent thermal conductivity was determined by a method equivalent to C-177. The test measurement was made by C-518 at a mean temperature of 75^{o}F. Mil-P-15280H, Paragraph 4.6.14, allows a choice of C-177 or C-518 to determine the thermal conductivity at a mean temperature of 75^{o}F. Results from the heat meter, C-518, were reported as thermal conductance Btu/hr.ft.^{o}F because a composite system, layers of different material, are normally reported as conductance. A homogenous system, e.g., foam or fiber glass, alone can be characterized as having a thermal conductivity. For comparison purposes, the conductance was multiplied by the test thickness, 1.10 inches, and the results reported as apparent thermal conductivity in Table 2.

Table 3

Tensile Measurement of Individual Samples

Product	Machine Direction Tensile, Lbs/in Width	Cross Machine Direction Tensile, Lbs/in Width
Product A	461	495
	511	544
	412	500
	434	500
	494	478
Average	462	503
Product B2	132	440
	219	423
	329	377
	445	379
	351	368
Average	295	397

Water Vapor Permeability - The water vapor transmission of thick materials C-355 was dropped by ASTM in 1982. The test method specified as its replacement is E-96, "Water Vapor Transmission of Materials". The test outlines two basic methods, the desiccant method and the water method. Duplication between the two test methods should not be expected. In testing facings for fiber glass, we normally use the desiccant method. The test conditions are 90°F and 50 percent humidity. The pans for holding the one-inch faced composite sample were fabricated from sheet copper and the dimensions were 10-inches square with a 3/4-inch ledge giving an exposed area of 8-1/2-inches square to the humidity differential. In testing Product A, both the desiccant method and water method were run. Because the specimens could only be sealed to the test pan in one orientation, the high humidity was on the fiber glass side for the water method. As shown in Table 2, the water method yielded a permeability value one-third lower than that obtained by the desiccant method.

Only the desiccant method was performed on Product B2 because of the limited number of test pans available. The desiccant method was chosen to maintain the high humidity on the facing side of the specimen. The results of a 21-day test were higher than expected, the water vapor permeability being greater than 0.3 perm inch in each case. When the Alpha 3267 MA facing material was tested alone in a five-inch pan, the weight gain was negligible over 21 days, representing a water vapor permeance far below 0.1 perm. The composite sample calculated between 0.48 and 0.51 perm inch depending on the time interval chosen.

The textured surface and the flexibility of the sample may have caused some faults in the sealing of the bees wax to either the facing or the test dish. Even the soldered joints of the test dish could have a pinhole which would not leak water under a preexamination, but would pass a vapor. The facing itself could have pinholes in its polyester film.

It should be pointed out that the facing samples tested originally and those employed in fabricating the materials delivered (and under discussion here) were from different lots. This difference could explain the discrepancies noted. Tests still in progress at this writing suggest this explanation.

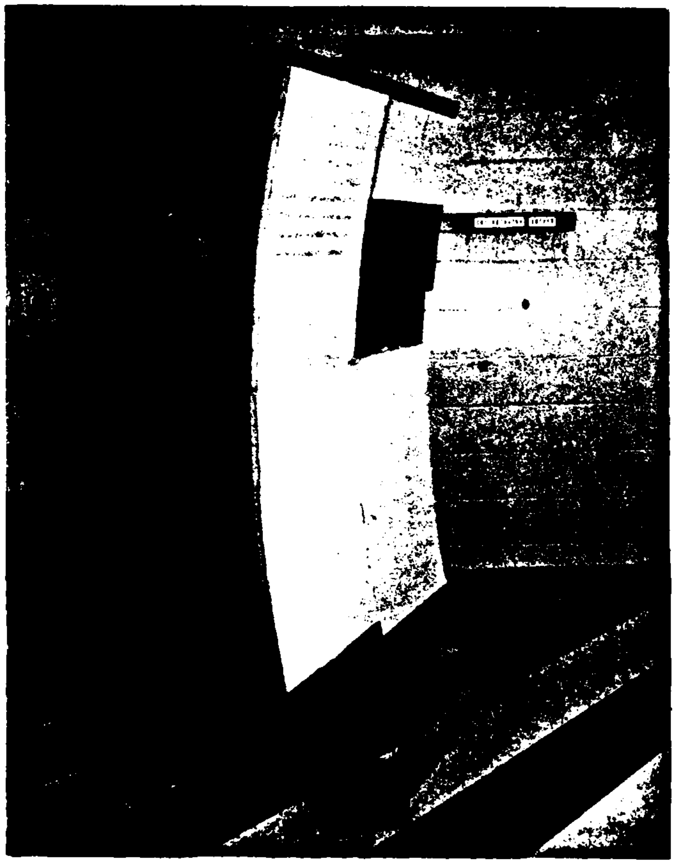


Figure 3. **Sub Hull Test Fixture** - A fixture was made to simulate the concave curvature of a sub hull. The dimensions are 6 feet vertical height and 4 feet in width. The sheet metal on the surface was bent to a 12 foot radius curve. Shown in the photograph are three large and three smaller one-inch-thick test pieces attached by Foster 30-04 adhesive.

Chemical Stability - A one square foot sample of each product was subjected to temperature cycling in a circulating oven. A total of six cycles over two days were completed; a cycle was 70°F for two hours and 175°F for hours. The samples were then conditioned at 70°F, 50 percent RH for two hours before being accurately weighed to calculate any weight loss that may have occurred. Highly favorable test results are shown in Table 2.

DETAILS OF DEVELOPMENT EFFORT

Factors Which Control Fiber Glass Product Performance Characteristics

Manville produces a broad range of fiber glass products, from lightweight mobile home insulation to high-density, rigid board products. Each product has its own unique characteristics fashioned into it by adjustment of the manufacturing parameters. An investigation was made during the first four months of the contract to determine the manufacturing parameters which have the greatest consequence on the desired physical properties and the interactions among the parameters in the ranges of interest. Table 4 lists the properties investigated and the second column enumerates the controllable manufacturing parameters to be correlated to those properties.

Requirements placed upon the first two listed properties, bending resistance and compression resistance, narrow the range of products to be considered to semi-rigid boards, 2 pcf to 6 pcf. Below 2 pcf the product's low compression resistance limits them to mainly roll and batt products. Above 6 pcf we would be in danger of exceeding the 0.75 pound per square foot specification in the composite product; also resistance to bending is substantially increased at this density. Table 5 has a presentation of selected one inch rigid and semirigid board products in the 2 to 6 pcf density range. The actual measured density, LOI (binder loss on ignition), and compression resistance are included. Because the compression resistance is dependent on other, unlisted factors such as fiber diameter, oven cure temperature and felting, the values will vary from day to day by an estimated 25 percent. The values listed were not based on samples from the whole population, but were from a random sample from a short production period.

The first listed property, compression resistance, was primarily affected by the type of fiberization unit. The rotary fiberization does not orient the fiber in the plane of the blanket as highly as does the pot-and-marble drawn and flame attenuated fiberization. Consequently, the rotary process felting demonstrated a compression resistance more than twice that for pot and marble (0.6 pounds per square inch versus 0.2 pounds per square inch) for comparable products in the 3 pcf density range. The other determining factors for compression resistance in order were density, LOI and fiber diameter. Both sample data and discussions with operating plant personnel were taken into consideration in setting this order. If the compressive resistance of two pounds per square inch is considered critical for the product, it appears a 4.5 pcf rotary product with 8-10 percent LOI would have a good chance of meeting that specification. The product submission made for the contract did not raise the LOI to the 10 percent range due to concern about the 1/4 scale test. The 10 percent LOI may later be proven to the practical, if deemed appropriate or necessary for adequate compression resistance.

In investigating the flexural rigidity, a formula related to the bending of a simple beam was used to provide comparison values between products.

$$EI = L^4P/192bD$$

L = sample length in inches
b = sample width (held at 6 inches)
P = load in pounds
D = deflection in inches

The sample length was manually set at 22 inches and the width cut to six inches. The thickness was assumed to be one inch for each product. A line load was applied at the center of the sample which was resting on a fulcrum at each end. The deflection and the load causing that deflection were recorded. Three samples from both the product machine direction (MD) and cross machine direction (CMD) were measured. The fiber length is generally oriented in the machine direction, therefore the strength is generally higher in the MD. In the flexural rigidity test (EI), fibers near the upper or concave side are in compression. Fibers on the bottom or convex side are under tensile stress. When the orientation of the sample is CMD, the fiber can separate perpendicular to the lengthwise direction and this generally requires less force.

From inspection of Table 6 we see the Armaflex foam, control sample showing only a small resistance (12.9) to a moderate bending. Moderate bending would be a one inch center deflection over a 22-inch fulcrum spacing. A bending resistance of 600 presented no problem in conforming to the 24-foot diameter curve of the test fixture. Two samples with resistance above 4,000 for EI did present a problem adapting to a curved surface. The samples generally showed a bias in their bending resistance. A bend in the sample was more easily accomplished when it was made in the CMD. In many cases, the force was reduced by half as compared to the MD. One product, 1001, was a roll crushed product wherein the compression resistance was reduced by half, the EI by two-thirds, and the tensile strength was basically unchanged. This could be one method of reducing the EI of a product. The controlling factors in EI appear to be LOI and density.

Tensile strength from reviewing Table 6 and from plant experience is most readily influenced by the fiber diameter. If needed, the specification value of 20 pounds per square inch could be met by a 3 pcf, 5 micron, 7 percent LOI fiber glass product alone, without facing or scrim. Once the fiber glass is incorporated into composite product the strength of the fiber glass becomes inconsequential to the overall strength of the composite.

The compression set was measured on seven fiber glass products. Four products showed zero compression set and three gave only a minor amount on the order of one or two percent. By comparison, the compression set of the 3 pcf Armaflex II control was 53 percent.

Smoke density test results E-662 were small for all tested fiber glass samples. The measured Dm values were under two even for samples with up to 8 percent binder. The Armaflex II samples gave a reading of 203. The test does not seem to be able to discriminate between binder levels in the fiber glass. However, this test is appropriate for discriminating between different types of facing which are employed in a composite system. The report section on facing has data on the facing smoke-test results.

Between the time when the samples were procured for the exploratory tests and the time of the plant trials for making the products submitted to the Navy, a change was made in the glass composition for all Manville products of the types with which we are dealing here. The absolute values of the data presented in this section of the report do not pertain directly to the present product line but the relationships would still hold.

Table 4

Factors Controlling Fiber Glass Product Properties

Property Investigated
Compression resistance
Bending resistance
Compression set
Smoke density
Tensile strength
Water absorption

Manufacturing Parameters
Binder formula
Fiber diameter
Percent binder addition
Product density
Type of fiberizing process

Table 5

Compression Resistance and Smoke Density of Tested Fiber Glass Product Samples

Product	Density Measured pcf	LOI Measured	Compression Resistance Lbs/sq in	E-662 Smoke Density Dm
Hullinsul	1.6	2.6	.14	0.4
SG 22	2.2	6.1	.12	1.8
Incombustible Hullboard	3.1	3.7	.59	0.3
650 Spin Glas	2.9	4.5	.69	1.4
830 Board	3.1	8.2	1.2	0.3
MAD Board(1)	3.4	16.2	1.4	
Zeston(2)	3.4	5.4	1.7	
1001(3)	3.4	4.2	.33	
Spin Glas 860	6.4	9.3	5.2	
Owens-Corning Navyboard	2.5	4.6	.75	0.3
Insulcoustic Navyboard	2.7	7.8	.88	0.3
Armaflex Foam	2.9	-	2.3	203

(1) A pot and marble fiberization product which would be expected to have a lower compression resistance than its rotary fiberization equivalent.

(2) A pot-and-marble product for which the fiber board is cut into strips, and the strips are rotated 90° and relaid on a backing, creating "bendability" and a high compression resistance.

(3) A mechanically crushed rotary board product which becomes a roll product after losing half its compression resistance.

Table 6

Tensile Strength and Flexural Rigidity of Tested Fiber Glass Product Samples

Product	Density	LOI	Fiber Diam. micr.	Tensile Strength MD Lbs/sq in	Tensile Strength CMD Lbs/sq in	EI MD	EI CMD
Hullinsul	1.6	2.6	4.7	13.0	9.1	100	71
SG 22	2.2	6.1	3.1	7.8	6.5	-	-
Incombustible Hullboard	3.1	3.7	4.5	9.5	9.3	310	142
650 Spin Glas	2.9	4.5	-	16.6	14.3	385	385
830 Board	3.1	8.2	4.7	31.4	25.5	596	360
MAD Board	3.4	16.2	-	98	59	4300	1710
1001	3.4	4.2	5.0	33	31.7	127	81
Spin Glas 860	6.4	9.3	5.0	41.7	41.7	4831	2395
Owens-Corning Navyboard	2.5	4.6	4.5	13.1	9.9	304	112
Insulcoustic Navyboard	2.7	7.8	3.4	9.9	9.4	218	284
Armaflex Foam	2.9	-	-	20.7	21.4	12.9	

Studies on Silicone Additives to Binder Systems

Polydimethylsiloxane polymers have been investigated and evaluated for water resistant properties. It is generally accepted by the textile industry that silicones can be applied to fibrous material to decrease hydrophilic or increase hydrophobic character of the material. The incompatibility of silicones with water results in excellent water-repellent properties. This repellency applies to liquid water only and not water vapor.

General advantages in the use of silicones to increase water repellency in industry are their excellent thermal and chemical stability at 150°C (302°F) in the presence of air, their chemical inertness, their resistance to dilute acids and bases, and their non-corrosiveness to metals.

Since the silicones were to be introduced into the product by spraying a single aqueous medium incorporating both the silicone and the phenolic binder, aqueous emulsions were investigated for this project. The dilutability of a silicone-emulsion and the chemical compatability with phenolic resin were the two major determinants for this study. The principal limiting factor seen in using a water-emulsion-type silicone is the hydrophobic nature of the emulsifier used to stabilize the emulsion. The reactivity of water emulsions with phenolic resin is also a limiting factor, thus decreasing the number of different water-emulsion-type silicones that could be evaluated for this study.

On the basis of these criteria, several silicones were selected for laboratory evaluation. Application by well-established laboratory aerosol-fogging techniques (known to yield bound fiber glass structures comparable to those obtained on production machines) did not prove satisfactory for silicone/phenolic combinations appropriate to this study. Rather than undertake to develop new techniques of uncertain relationship to actual product operations, it was deemed best to perform the pilot experimentation in a bona fide plant trial. The assured reliability of results was thought to more than offset the added complexity and cost of this originally unplanned plant trial.

Against this background, three silicones were selected for evaluation in a plant trial in Corona, California. These silicones were mixed directly into the phenolic resin binder tank at known concentrations. The total solids content of the batch was maintained at the level used under normal working conditions. While maintaining total solids constant application efficiency was held at about 85 percent. Eleven different board products were produced (in accordance with a statistical design) to evaluate the repellent behavioral aspects in reference to different types of silicones and their concentrations, while maintaining constant phenolic levels through each of several subsets. Differences in the silicones used were in their emulsifier, chemical functionality and the phenolic compatability. Compositions of the eleven products from the Corona trial are presented in Table 7, along with results of tests described in the following section of the report.

Test Methods Used to Evaluate Water-Repellency

Water-Absorption Test I (Mil-P-1528-OH/4.66)

4.6.6 - Water Absorption

4.6.6.1 - Specimens - Test specimens shall be 4 by 4 inches square or semicylindrical sections (tubular form cut in half longitudinally) 6 inches long in the thickness furnished. The specimen may have the skin on top and bottom, outer and inner surfaces, or on only one of these surfaces, as specified in 6.2.

4.6.6.2 - Procedure - Specimens shall be submerged in distilled water at room temperature, 70°F to 80°F, 2 inches below the surface of the water and subjected to a vacuum of 25 inches of mercury for 3 minutes. Release the vacuum and allow the specimen to remain submerged for 3 minutes at atmospheric pressure. Remove the specimen, allow to stand on end to drain for 10 minutes and blot lightly with paper towels. Values of each of the three specimens shall be calculated and reported in terms of pounds of water gain per square foot of skinless surface.

Water-Absorption Test II (Aerospace Industry Test) - Specimens were prepared as in Test I, above. However, the samples were immersed six inches below the surface of water at room temperature and at atmospheric pressure. Duration of immersion was six minutes. Post-immersion procedures were as prescribed for Test I. We believe Test II represents more accurately the water absorption hazard to which the insulation product may be exposed in service than does Test I.

Aerospace Wick Test

7.11.1 - Wicking of Material as Received

a. Cut six 1 by 6 inch specimens from the batting mateial with the 6 inch length in the direction of the roll. Cut six similar specimens with the 6 inch length parallel to the width of the roll.

b. Fasten loosely, with fine wire, six specimens (three cut with the roll and three cut across the roll) to a grease-free 0.035, 4 by 4 mesh galvanized wire screen and position this assembly in an upright position so that the ends of the specimens touch the bottom of the container. The specimens must not touch each other or the sides of the container. Pour distilled water into the container to a height of 1 inch. Maintain the temperature of the water at 120 + 5F. Note degree of wicking every 24 hours. Materials must not wick to greater than 1/4-inch above the water line in 168 hours.

Aerospace Surface-Wetting Test - Surface wetting is determined from the wicking specimens. Surface wetting (not considered as wicking) cannot extend more than one-inch above the water line.

Wetting is defined as a condition where the water has penetrated into the insulation and fills the spaces between the fibers. Beads of water are not to be construed as a condition of wetting. The formation of beads of water on the insulation surfaces may be due to condensation and indicates water repellency.

Test Results and Discussion

Results of water absorption and wicking tests on the eleven products made in the Corona plant trial are presented in Table 7.

Table 7

Absorption and Wicking Test Results

Sample ID[a]	Water Absorp.[b] Test I Lb/ft^2	Water Absorp.[b] Test II Lb/Ft^2	70 Hr. Wick Test	168 Hr. Test	Surface Wetting
1. 4.0/0.0 -	2.2	2.3	F[c]	F	P[d]
2. 4.0/0.5 A	1.3	0.07	P	F	P
3. 4.0/0.5 D	0.9	0.03	P	F	P
4. 4.0/1.0 C	0.9-	0.01	P	F	P
5. 4.0/2.0 D	0.8	0.01-	P	P	P
6. 4.0/2.0 C	0.8-	0.04	F	F	P
7. 8.0/0.0 -	2.2	2.1	F	F	P
8. 8.0/1.0 A	1.7	0.01	P	F	P
9. 8.0/4.0 A	1.7	0.02	P	F	P
10. 8.0/8.0 A	1.8	0.42	F	F	P
11. 6.0/2.0 A	1.4	0.01	P	F	P

a. Sample identifications include, in order: product (trial) number, targeted phenolic content in percent by weight, targeted silicone content in percent by weight, and silicone type designated by code letter. Silicone D is classed as a reactive silicone. Silicone B was not employed in the Corona trial.

b. Water-absorption data presented here are calculated from experimental measurements in accordance with our interpretation of the wording of Section 4.6.6 of MIL-P-15280H. Thus, "skinless surface" of 4 x 4 x 1 inch sample with facing material on one 4 x 4 inch face is calculated as 32 square inches (0.22 square feet). If one calculates on basis of "area in the plane" of the sample, the area is 16 square inches and the water absorption values are twice those in the table. It should be noted the values reported in the March and May monthly progress reports were based on the figures on the projected plane area and thus were twice the values cited in Tables 7 and 15.

c. F designates failure to meet test criterion.

d. P designates compliance with test criterion.

As would be expected, none of the materials from Corona fared well in Water Absorption Test I. However, the effect of the silicones is apparent, especially at the lower phenolic contents.

The silicone-free controls performed no better in Water Absorption Test II than in Test I. On the contrary, the silicone-bearing materials were better by more than an order of magnitude in Test II (relative to Test I) in all but one case, that of Product No. 10. In comparing Test II data it is clear that this sample, with very high silicone content (eight percent), is less repellent than corresponding samples with lower silicone content. This is attributed to the backwetting influence of the emulsifying agents employed in the silicone formulation.

The qualitative (pass/fail) data on wicking are generally in harmony with the water absorption test results. Surface wetting tests were inconclusive.

Overall, the data of Table 7 suggest that one-to-two percent silicone combined with four-to-six percent phenolic will give best water repellency and that the reactive silicone, D, is the one of choice where it can be used. Where it cannot, Silicone A would appear to have an edge over Silicone C.

Chemical instability was observed with a great majority of the reactive silicones studied in mixtures with phenolic binder systems in the course of the program. The emulsifiers used in most reactive silicone formulations are cationic, and, therefore, promote flocculation when mixed with the phenolics. With few exceptions, such as Silicone D, the flocculation is so rapid and severe as to obviate their use in a production situation. On the other hand, the majority of the non-reactive silicones were chemically compatible with the phenolic resins employed.

Fiber Glass Scrims

A decision was made early in the program to undertake creation of a composite product which could be attached to a 33 foot diameter curved hull surface without the use of studs. A tough covering sewn on the insulating blanket could make this feasible. Sewing of a tough temperature-resistant fabric on high-temperature fibrous or particulate insulations has been employed by Manville on such products as Min-K and the Quilite space shuttle insulation. However, none of these products required a vapor proof surface. In the present case, the vapor proof surface could not be stitched on the product without impairing its effectiveness. The surface to be sewn on must be tough, fire resistant, lightweight, and low cost. Fiber glass scrims were chosen because of the features mentioned.

When the scrim is sewn to both sides of the fiber glass board by sewing through the board, the system strongly resists delamination. A second benefit is that tensile strength (in the plane) increases from 15 pounds to above 100 pounds per square inch due to the strength added by the scrim.

When scrims were first considered, scrims without a set finish to hold the threads in place were considered. These were fire resistant, but the weave easily separated, especially after being cut to size for sewing on the surface of the fiber glass. To keep the threads in place a set finish is applied. The finish is typically a PVA or PVC emulsion, which imparts a modest flammability to the scrim. In comparing two finishes - PVA and PVC, the PVC shows a lower flame spread in a small scale fire test.

Scrims are made with either a plain or leno weave. A leno weave employs a twist of twin weft yarns between each pair of warp yarns. Table 8 gives a list of scrim samples obtained for testing. For sewing on a fiber glass board, our plant personnel preferred a material with lower weave count and with a finish.

The scrim chosen was the J. P. Stevens 1350/38 leno 6 x 6 weave with a PVC finish set. The advantages of the choice were ease of sewing and strength; a possible disadvantage was relatively few yarns for an adhesive to grip.

Scrim Sewing

Manville has facilities for sewing insulations at its Manville, New Jersey plant. Three "fixed" scrims, including the J. P. Stevens 6 by 6 and a 10 by 10 scrim without a set finish were sent to Manville for preliminary sewing trials on either an incombustible hullboard or an 830 board which had twice the compression resistance of the incombustible hullboard, 1.2 pounds per square inch versus .6 pounds per square inch. The tests were to determine whether the plant personnel found it easier to stitch the stiffer fiber glass product. They did not voice a preference for the stiffer product, so this was not a factor in future sewing requests.

TABLE 8

Fiber Glass Scrim

Company	**Scrim Type**	**Weave Count**	**Wt. oz/yd.**	**Breaking Strength MD x CMD lbs/in**	**Finish Set**
Claremont	1653 Leno	8 x 8			No
	1659 Leno	10 x 10			No
	7603 Leno	5 x 5			No
Baytex	Plain	5 x 6			Yes
Hexcel	1658 Plain	10 x 10			No
Burlington	1658 Plain	10 x 10	1.6	80x70	No
	1659 Leno	20 x 10	1.6	65x70	Yes
	1631-38 Leno	15 x 14	2.2	90x100	No
	1631-632 Leno	15 x 14	2.2	90x100	Yes
J. P. Stevens	1659/36 Leno	10 x 10		80x75[1]	Yes
	1350/38 Leno	6 x 6	3.5	130x120[1]	Yes

(1) Manville R&D measured value, the value refers to machine direction and cross machine direction.

A total of seven boards were sewn with a scrim on both sides. The only scrim which posed a problem was a 10 x 10 from Claremont which did not have a finish set. The scrims which were sewn on and presented no problem were; Burlington 1631/632, Burlington 1658/36, and J. P. Stevens 1350. One of the 24 inch by 33 inch pieces was stitched with the fiber glass on a bias, so the scrim would stretch when the piece was bent with that scrim on the outside of the bend. While this could be a useful technique where sharp bends are required, only an 0.18 inch stretch is needed for a three foot piece to fit a 33 foot diameter curvature. The seven pieces with a scrim sewn on both sides were used in the subsequent adhesive studies.

A two-inch by two-inch quilt was the pattern used for the seven trial pieces. The stitch pattern affects the handling and appearance of the product when installed. When stitched in a two-inch by two-inch cross hatch pattern, the facing will show a quilted appearance when attached to a concave surface.

The pattern for the final submission completed in May 1983 was a 3 inch by 3 inch pattern, which is less expensive. The ends of the threads used for the backing stitch were not tied off after sewing. The ends were tied off by hand after the pieces were shipped to Manville R&D for the application of the facing. If the edges were finished on the ultimate, fully-developed product, the sides of the pieces would be scrim covered and the thread would be stitched back into the piece. One sample piece of this description was made. On all the other sample pieces, a row of stitching was made within one inch of each edge of the fiber glass piece and extended the entire width of the piece. This prevents the edges from lifting up. To reduce the cost of sewing even further, a three-inch horizontal only stitch was attempted. It was decided the appearance on a concave surface was not as desirable as a three-inch square pattern. The samples when bent 90 degrees tend to bend on a line with the stitching. The stitch pattern should be made to conform with any sharp bending required.

The thread used in the stitching is an E-18 teflon coated glass thread. The thread is not waterproof and the teflon would give off noxious gases if burned. A silicone-polymer-coated thread would offer the advantage of water rejection and would not give off florides when burned. A vendor such as Fil Tec could coat and cure a silicone polymer on the thread. A silicone oil coated thread is presently available but an oil is less desirable than a polymer. Use of such sewing threads would require some development work, so the current program was limited to use of threads with which the operators had experience.

Facing Materials

The facing for the composite product has severe requirements for durability, paintability and impermeability to water vapor. The last requirement necessitated the inclusion of a vapor barrier. Common vapor-barrier materials would include rubber, latex, and Mylar films. The thickness of the Mylar film can vary from 1/2 mil to a relatively thick 5 mils. The thicker films may have a lower probability for pin holes but would contribute more fuel to a flame.

Sixteen companies were contacted in the search for suitable vapor-proof laminates. A positive response was received from seven companies. Their submissions are listed in Table 9. Two of the submissions were typical fiber glass board coverings. Only those from Claremont, Alpha and Lamtec were considered likely candidates. The Lamtec sample, specially made in a pilot run, was similar to the 3267 MA facing from Alpha. Their effort resulted in a product with good appearance, but the Mylar delaminated from the fiber glass Navyboard cloth during adhesion tests. In comparing Alpha's 3267 polypropylene laminate to its 3267 MA aluminized Mylar, the Mylar was found superior in the facing adhesive tests. This narrowed the candidates to Alpha 3267 MA and Claremont 2S14 R Tuffskin.

A comparison was made (see Table 10) among four facings for smoke density. One facing, the Claremont Tuffskin, gave a smoke density significantly higher than the other three, but not so high as to disqualify its use. We should note here that the sample with Tuffskin facing included substantially more adhesive than did the other samples.

A water vapor transmission test on the Alpha facings 3267 and 3267 MAU was performed in accordance with ASTM E-96, the Desiccant method. There was no measurable weight gain over a 21-day period in either case.

When comparing the strength of five facings in Table 11, the Mullen burst strength is in the range of 630 to 708 psi. The burst strength of the facing did not differ significantly from each other. The tensile strength ranged from 620 pounds per inch to 1020 pounds per inch showing the very large contribution the facing can make to the tensile strength of the composite.

TABLE 9

Facings

Company	**Description**
Burlington	3732 Fiber Glass Navy covering
Hexcel	332 Fiber Glass Navyboard covering
Newtex Industries	Rubber coated heavy weight fiber glass
Claremont Company	2S14R Tuffskin 2-mil Mylar with fiber glass laminate on both sides
Alpha Associates	3267 - Polypropylene laminate on Navyboard cloth
	3267 MA 0.5 - mil aluminized Mylar laminated to Navyboard cloth
	3267 MAU - Treated 0.5 Mil aluminized Mylar laminated to Navyboard cloth
Lamtec Corp.	Aluminized Mylar on Marine Board facing
J. P. Stevens	2025/48 Aluminum foil laminated to heavy weight fiber glass cloth

TABLE 10

Facing Smoke Density Test[1] ASTM E-662

Sample Facing	Adhesive Wt. LAMX 235 (gm/sq. ft.)	Maximum Optical Density Flaming Condition	Non-Flaming
Hexcel Navyboard	5	5	12
Alpha 3267 MA	2	11	11
Alpha 3267 (polypropylene laminate)	2	8	15
Claremont 2S14R	10	46	51

(1) Samples were composites made from 3 pcf Incombustible Hullboard, adhesive and facing.

TABLE 11

Strength of Facings

Facing	ASTM D-777 Mullen Burst (psi)	ASTM D-828 Tensile Strength MD (lbs/in. width)	CMD (lbs/in. width)	Facing Wt. (gm/sq.ft)
Claremont 2S14R	708	1020	620	60
Hexcel 332	635	1020		41
Alpha 3267	665	890	765	45
Alpha 3267 MA	649	655	1100	48
Burlington 3732	630	970		44

Facing Adhesives

The function of the applied adhesive is to attach the facing to the sewn scrim durably and in such manner that it will follow the contours of the insulation when attached to a curved surface. The grip should be tight enough that the sewn piece does not separate from the facing with a vigorous shake. The principal strength of the attachment is to the mesh scrim and not to the glass fibers of the blanket. A primary limitation was that the adhesive was to be water based to be compatible with requirements for application in a plant. The adhesive should not require elevated temperature for curing.

The requirement posed to adhesive companies was two-fold - an adhesive which would give the proper attachment and also offer low smoke and low flammability potentials. The surfaces to be attached were fiber glass scrim to fiber glass weave and fiber glass scrim to aluminized Mylar film. It was anticipated no single adhesive would be suitable for both applications. The adhesives tested and the companies who supplied them are listed in Table 12.

The first applications were made by Myer rod (a rod with fine circular grooves) on the facing. The loading by this method was only four grams per square foot on the Mylar laminate. For a trial of the adhesive, a three inch by three inch area was adhered to a plain weave scrim (J. P. Stevens 1659). After drying, the scrim was pulled away from the facing in an Instron tester at twenty feet per minute. A sudden failure of the scrim occurred at four pounds' pull due to the 180° bend in the scrim threads, cracking and individual breakage. The scrim itself breaks at a 100 pound pull when subjected to just tensile force. This adhesive test did not correspond to actual use conditions so it was deemed unsuitable.

The test ultimately used to rank adhesives was to apply the facing to a scrim-sewn piece of blanket and then manually pull the facing off at a right angle to the scrim-sewn piece. The adhesive was normally allowed to set for 24 hours before the facing was torn away, but it was observed that the strength of many adhesives continued to improve up to three days after application.

An evenly laid film of adhesive of up to 10 grams per square foot did not tightly grip the fiber glass scrim. However, by experimentation we found that a dot pattern using 10 grams per square foot gave the adhesives a much better grip on the scrim threads. The pattern was laid down with the help of a template, a 20-gauge metal sheet with 1/4-inch diameter holes punched at regular intervals. A ranking of adhesive bonding was formed by applying each adhesive to a six inch by three inch piece of both the Claremont and Alpha facing and then pulling the sample piece from the scrim sewn piece. The rankings can be found in Table 13.

A small scale fire test which Manville uses to test facing flammability and smoke generation was employed to rank the flammability/smoke of the water-borne polyvinyl acetate (PVA) and latex adhesives. The test is based on ASTM D-777, which is a discontinued ASTM test.

For the test, the adhesive was applied in a dot pattern on the Claremont facing. The six-inch by three-inch piece was held (with six-inch dimension vertical) by clips over a 1-1/2-inch Bunsen burner flame for thirty seconds. The burn was carried out in a glass faced chamber which held the smoke until the door was opened. In Table 14, in which test data are presented, we see two of the adhesives, Northwest 404 and National Starch 40-0857, allowed the flame to climb up the full length of the facing, and showed highest smoke generation, as well. In Table 13 we also note each was the strongest adhesive for one of the facing materials. Because of the flame spread neither of these adhesives was chosen.

The adhesives chosen, General Latex, 3S-716A and National Starch 72-6800, represent a compromise among the desired properties. The General Latex adhesive is a pliable latex with good water resistance. It was used for the woven fiber glass surface of the Claremont facing; its adhesion to Mylar was not as good. For the Mylar surface of the Alpha facing, the choice was National Starch 72-6800, a more rigid and less water resistive PVA adhesive. (In general, such adhesives will dissolve slowly in liquid water). Each of the adhesives will withstand handling in installation and sharp bending, but the facing can be separated off the scrim-sewn piece by a strong hand pull.

Table 12

Company	Adhesive	Comment
National Starch	40-0857 72-6800	UL approved, Fluid Viscous Fluid
Northwest Chemical	404 385	UL approved
Swift Company	10-020 6782HS DA3-6782	UL approved
Manville	LAWX 235	UL approved
General Latex	3S-716A 10S-733	UL approved
Foster	30-04	Mil-A - 3316B

Table 13

Ranking of Adhesive Bonding to Scrim Sewn Board

Claremont 2S14R	**Alpha 3267MA**
Northwest 404 (highest)	National Starch 40-0857
National Starch 72-6800	Northwest 404
General Latex 3 S-716A	National Starch 72-6800
National Starch 40-0857	Northwest 385
Northwest 385	General Latex 10-S-733
General Latex 10 S-733	Swift 10-020
Swift 10-020	General Latex 3-5-716A
Manville LAWX 235	Manville LAWX 235
Swift DA3-6782	Swift DA3-6782
Foster 30-04 (lowest)	

Ranking based on 10 gm/sq ft dot pattern

Table 14

Ranking* of Adhesive on Flame Height and Smoke Density

(Small Scale Fire Test -

1-1/2-inch Bunsen Burner Flame 30 Sec.)

Adhesive	**Smoke Generation****	**Flame Height Up Facing**
Northwest Chemical 404	(highest)	6"
National Starch 40-0857		6"
Swift 10-020		4"
Northwest Chemical 385		4"
National Starch 72-6800		4-1/2"
General Latex 10-5-733		3-1/2"
Manville LAWX 235		4"
General Latex 3-S-716A		3"
Swift DA3-6782		3"
Foster 30-04	(lowest)	2"

* Ranking based on 10 gm/sq ft dot pattern.

** Samples listed in order of decreasing smoke generation in this test.

PRODUCTION OF PRODUCTS FOR SUBMITTAL

Manufacturing Trial at Richmond, Indiana

A production trial on one of our full size rotary fiberization units was completed in May. Four experimental, 3 pcf, water repellent, fiber glass board products were made, each with its unique combination of binder and silicone added. The products were labeled A, B1, B2 and C. Table 15 gives a product description and physical test results.

During the trial, the product changeover needed to be made efficiently because of the several thousand pounds per hour capacity of the unit and the requirements of only 500 square feet of each experimental one-inch thickness product. In the trial plan it was proposed to build in a binder distribution showing a bias of a higher concentration on one side as compared to the other. It was thought the lower, approximatly 4 percent, binder side would stretch more easily and be used on the tension (convex) side of the hull-conforming system. The higher binder side, 6 percent, would be fixed to the facing on the inside (concave) side of the system. Experimental confirmation of this theory was not effected because of the time constraints. A second directive was to set the fiber diameter 0.5 microns larger on the bottom or higher binder side, again with the intention of introducing asymmetric bending resistance. This was attempted but the action did not consistently bias the fiber diameter in the manner requested in this first trial of the concept. The overall average fiber diameter requested was 5 microns. This was achieved in Trial A and B1 but the diameter had drifted to 4 microns at the time Product C was produced. The 4- to 5-micron size is the normal size average of fiberization on the machine employed. While there is significant flexibility here, a request for fiber above six microns may cause some collection difficulties. The several products issued from the machine as flexible boards, nominally one inch thick. These products were cut into two-foot by three-foot sections with the three-foot dimension in the CMD to minimize resistance to lengthwise bending.

One product run, B1, did experience temporary manufacturing upset. The quality of the final B1 product reflected this, as can be seen in the physical property data. The tensiles were below 10 pounds per square inch and the compression resistance, 0.64 pounds per square inch was the lowest of the four products.

B1 was rejected in the final selection process. This left three products from which two choices would be sent to Manville, New Jersey, for scrim sewing. Product B2 was one choice because it was the only remaining product incorporating a reactive silicone. Of the remaining two prospects, A and C had the same compression resistance, but A had a better water absorption, 0.01 pounds per square foot, and was therefore selected. Two hundred and fifty square feet of each product selected were shipped to Manville to have a scrim sewn on both sides.

Table 15

Richmond Experimental Silicone Additive Fiber Glass Board Production Trial

Prod. Label	Sil. Add. %	Phen. Bind. %	Prod. Dens. pcf	Tensile MD Lbs/ sq in	Tensile CMD Lbs/ sq in	Comp. Resist. Avg. Lbs/ sq in	Water Absor. Lbs/ sq ft	Fiber Diam. Micron
A	2	5	3.3	23.2	16.6	.84	.01	5.0
B1	1	5	2.7	9.6	7.9	.64	.04	5.0
B2	3	5	3.1	11.0	10.3	.85	.02	4.4
C	2	5	2.7	13.5	11.1	.85	.04	4.0

* Submerged under 6-inches of water for 6 minutes at atmospheric pressure.

Product Facing Application

Quilted (scrim sewn) batts (flexible boards) were shipped from the Manville, New Jersey plant to the Research and Development Center at Denver for facing. For ease of application (e.g., to assure complete coverage and maximum contact of adhesive), oversize sheets of facing were applied to the two-foot by three-foot batts. The application of the product facing was accomplished using a dot pattern of adhesive on the backside of the facing. The adhesive/facing combinations were General Latex 3-S-716A/ Claremont 2S14R and National Starch 72-6800/Alpha 3267 MAU. Both of the adhesives were viscous, above 2,000 cps, which was advantageous in holding a droplet apex which was necessary to cover the surface of the threads in the scrim during assembly.

The adhesives were applied using a perforated metal template and roll coating the adhesive through the 1/4-inch holes. A 20-gauge, N330 perforated steel sheet from Diamond Perforated Metals, Inc. was used. Every other row of 1/4-inch holes was covered by masking tape to create a sheet with a 20 percent open area. This application gave an adhesive coating of 10 to 15 grams per square foot, depending on how thickly the roll coating was applied. The target dry weight for the adhesive was 12 grams per square foot of facing.

After the adhesive was applied to each piece of facing, it was laid on the darker side (higher binder side) of the scrim-sewn batt. Each piece was weighted by a board, one pound per square foot, to maintain good contact between the facing and the scrim. Even so, the adhesive in the area of the stitch rows did not contact the scrim because of the quilting effect of the stitching.

The next day the board was removed and the piece allowed to finish drying with the facing side down. Because the board and the facing do not allow vapor passage, the center of the piece had adhesive which had not totally dried in 24 hours. The excess facing was then trimmed with a knife and the composite insulation packaged for shipment.

CONCLUSIONS

Two composite insulations incorporating a semirigid fiber glass board were produced which met or exceeded ten of the thirteen specified properties. A fourteenth specified test, the 1/4-scale fire test, was not included in the test results because it is to be run by NRL at a later date. Measured properties which were significantly better than the specified properties were:

	Measured	**Specification**
Compression Set	0-1%	$\leq$25%
Dimensional Change	0-.1%	$\leq$10% of original length
Tensile Strength	350-480 lbs/in^2	$\geq$20 lbs/in^2
Apparent Thermal Conductivity	.23 - .24 Btu.in/ $hr.ft^2.^oF$	$\leq$.30 Btu.in/ $hr.ft^2.^oF$

Three specified properties were not met and comments on each of the three are included here.

One test, the water vapor permeability, unexpectedly gave results which do not fall within the property specifications. The Alpha facing, when tested by itself in a 5-inch dish by the desiccant method, gave no perceptible weight gain, translating to a permeability of very substantially less than 0.1 perm. But when a composite insulation, B2 with that facing, was tested in a 10-inch square pan, there was a weight gain equivalent to a calculated water-vapor transmission rate (WVTR) of .5 perm inches. This result may be attributed to lot-to-lot variation of the facing material or to problems with the test when applied to thick, rough-surfaced materials. It appears that the test requires practice to achieve the low WVTR values of which a vapor-proof facing is capable. In this vein, it is interesting to note that Battelle (in its Summary Report under Contract N00024-79-C-5331) reported a WVTR of 0.9 perm inches for the Armaflex II, while the manufacturer has reported 0.17 perm inches. Product A, which was tested by both the water and desiccant method, gave results of 0.6 perm inches and 0.9 perm inches respectively. The comments made on Product B2 would also pertain to Product A.

Our comment on the second test, compression resistance, which did not meet the property specification of 2 pounds per square inch at 25 percent compression, is to encourage an examination of the property specification. It is possible to increase the compression resistance of the fiber glass product to the specified range by increasing the fiber density to 4.5 pcf and doubling the phenolic binder content to 10 percent, but this action increases product areal density by 25 percent and the organic content substantially. This latter could affect the 1/4-scale fire test adversely. Both of our products should be examined for the appropriateness of their compression resistance value, 0.9 pounds per square inch, to the intended application. The specification after consideration might be reduced to one pound per square inch, or even lower, without adverse impacts on product performance.

For water absorption, the test as set forth in Section 4.6.6 of Mil-P-15280H probably cannot be passed by any open cell or fibrous product. If the procedure requirements are changed to involve only simple immersion under water at atmospheric pressure, products of the type we have developed are capable of meeting the 0.1 pounds water per square foot of skinless surface. It would be reasonable to conclude that the test at atmospheric pressure is more meaningful in relation to the service environment anticipated for the insulation product. In comparing our two products, it is noted Product A, with the coarse Claremont facing, absorbed more water than Product B2, with the finer Alpha facing. It should also be noted that to date we have made no effort to introduce water repellency into the facing materials or into the scrims or sewing threads. We are confident that treatment to introduce this characteristic will substantially reduce water sorption of the composite products.

The proposed composite insulation offers its greatest advantages over other fiber glass insulations in the intended application through its capability for attachment to the hull surface using adhesives and its water-repellent nature. Welded studs are not necessary for its permanent affixture, and debilitating water absorption would not be anticipated in submarine service. In comparison with other types of insulation, those developed in the subject work offer the superior fire resistance, low smoke generation, demensional stability and recovery, low density, and low thermal conductivity associated with fiber glass insulations.

One further conclusion of particular importance is that the materials submitted to the Navy represent the state of development reached in a nine-month development effort, and do not by any means represent the ultimate achievable in reaching an optimum combination of performance characteristics. The program has provided the foundation for such further development as may be deemed necessary for submarine hull insulation or for other Naval applications. It is suggested that the special combination of typical fiber glass characteristics with water repellency, vapor barrier, and high resistance to delamination (stud-free mountability) will be attractive for solution to a number of Naval insulation problems.

RECOMMENDATIONS

A potentially valuable fiber glass insulation concept has been developed and embodied in product samples submitted to the Navy. Several steps toward refinement and full exploitation are recommended.

1. Assessment of true product performance requirements appropriate to a fire-resistant, anti-sweat, fiber-glass submarine hull insulation, vis-a-vis, the 14 characteristics called out in the product specification targets established for the completed program.

2. Refinement and extension of the specifications to achieve a complete description of the product required.

3. Assessment of Products A and B2 against the resultant set of specifications.

4. Studies to resolve uncertainties re water-vapor permeability test results and to establish transport characteristics of condensed water in water-repellent fiber glass structures of the type developed here.

5. Further development efforts as necessary to bring the fiber glass products into full compliance with the set of specifications generated in earlier steps in this sequence.

6. Development and test (demonstration) of the complete insulation system and installation and maintenance procedures.

7. Studies to identify other applications in which products of the type developed here (with or without adaptation) might be beneficially employed by the Navy.