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CARBORUNDUM

Research and Development Division · The Carborundum Company · Niagara Falls, New York

THERMAL/ACOUSTICAL AIRCRAFT INSULATION MATERIAL

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Final Technical Report
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Prepared Under Contract NAS 3-14482

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I. INTRODUCTION

On March 1, 1975 the development work of the Thermal/Acoustical Aircraft Insulation Material was reactivated in the Research and Development Division of The Carborundum Company. The main objective was to improve the acoustical property of the Fiberfrax® foam developed under contract NAS 9-13641 sponsored by NASA.

The final Fiberfrax® foam developed under NAS 9-13641 containing about 16% AAA glass fiber displayed excellent thermal protection properties but fell short of the desired acoustical properties. It is the objective of the current contract, NAS 9-14482, to improve the acoustical attenuation of the Fiberfrax® foam based on results generated from the first year's efforts.

The approaches used to improve the acoustics of the foam include:

- Optimization of fiber blend composition and
- Modification of the foam fabrication process to achieve a well dispersed foam structure.

Fiberfrax® foams with the acoustical properties meeting the target values have been prepared. These foams contain 40% to 50% of AAA glass fiber.

In the subsequent sections discussions will be devoted to process modification, material selection and product characterization.

II. SUMMARY OF RESULTS

During the contract period beginning March 1, 1975 and running through May 31, 1975 our efforts were focused on improving the acoustical properties of the low density Fiberfrax® insulation specimens. Samples prepared in the contract period were characterized both in Carborundum and NASA for physical, thermal, and acoustical properties. Summarized below are the achievements of this contract period:

A. A two step fabrication process, formation of low density foam and pressing to desired thickness and density, was developed. The modified process enabled us to produce a well dispersed foam specimen containing a higher percentage of ultra fine glass fiber (AAA microglass) for acoustics improvement purpose.

B. Two formulations of Fiberfrax® foam using 40% AAA glass fiber have successfully achieved the acoustical requirements of the contract.

C. Flame impingement tests of the above two formulations have shown satisfactory results. Most importa[illegible], the sample using Fiberfrax® short staple in its composition provided the best results in both tests -- a composition yielding an optimum balance of required properties.

III. FUTURE WORK

Thus far, the development work carried out demonstrates the feasibility of the process to produce a light weight thermal/acoustical aircraft insulation material. Incorporated in the structure of this insulation material are blends of Fiberfrax® fibers and AAA glass fibers. To further optimize this process effort should be continued to develop the ideal composition which will yield the maximum balance of thermal/acoustical properties that can improve the safety of future aircraft. It is proposed that the efforts should be devoted in the following areas:

A. Optimize the Fiberfrax®-glass fiber ratio to achieve the best balance between the thermal/acoustical properties while improving the mechanical strength of the specimen.

B. Determine if the need exists for an additive to impart strength to the sample.

C. Produce sufficient quantities of Fiberfrax® foam specimens for fuel fire test at NASA.

D. An engineering process study to produce foam in large quantities consistently and economically.

IV. DISCUSSION OF WORK

A. Process Development

The fabrication of Fiberfrax® foam was accomplished by dispersing Fiberfrax® fiber with a foaming agent using water as the dispersing medium followed by removal of water and proper heat treatment. The overall procedure consists of three basic steps:

1. Mixing

   In this step the mixture of dispersing fibers and water was prepared and was mechanically stirred in a blender at relatively low speed. The purpose of this operation was to break up the fiber clusters and to insure homogeneous distribution in the foaming step.

2. Foaming

   To the slurry from "1" above a suitable surface active agent (foaming agent) and an organic binder were added. The mixture was then agitated to cause a foaming action by a high speed blending action or a constant flow with a circulating pump. In either case, a thick heavy foam similar to a shaving cream was formed when the proper conditions were used.

3. Drainage and Cure

   The water content in the foam was removed prior to final heat treatment. This was done by pouring the foam into a perforated metal mold and allowed to drain. The mold was then placed in a mechanical convection oven. The objective was to cure the organic resin so as to produce the well bonded foam structure. It was with this general

process that a low density Fiberfrax® structure was developed. To meet the objective of improved acoustics process modifications were made and are discussed in the following sections.

B. Material Parameters Evaluated

1. Fiberfrax®

The dispersion fiber initially evaluated in producing a light weight thermal/acoustical aircraft insulation material is a ceramic fiber, Fiberfrax®. Several grades of Fiberfrax® are available and their properties are shown in Table 1.

Those fibers selected for evaluation to improve the acoustic attenuation include long and short Fiberfrax® together with HiFi fibers. The latter fiber has an average fiber diameter of 1.6μ which is favorable for improving acoustics while still imparting thermal properties. The former two fibers were extensively evaluated in the process development period and are important to the thermal and structural properties of the insulation material.

2. Glass Micro-Fiber

Two grades of Johns-Manville Micro-Fiber, AAA (0.5 to 0.74μ) and AA (0.75 to 1.49μ) were evaluated as components of the Fiberfrax® foam. Improvements in acoustical properties were achieved by increasing the percentage of these fibers from previous 16% to 50%.

3. Surface Active Agent

Surface active agents were used to facilitate wetting and foam formation of the Fiberfrax®/water slurry. Emulsifier AH-861 and Sulframin® AOS slurry were selected for

. Density variations

To correct the situation a modified fabrication process was developed. A low density foam was made first and given a low temperature heat-treatment at 125°C for 30 minutes. This was followed by pressing the foam to the desired thickness and density before the final heat-treatment.

Samples fabricated by the new process showed markedly improved uniformity. The density of the samples produced ranged from 0.8 to 0.95 lbs/cu.ft, with the majority of the samples falling between a density of 0.85 to 0.9 lbs/cu.ft.

Fiberfrax® foams with different Fiberfrax® and AA glass fiber ratios as well as different dimensions were fabricated. The fabrication details are discussed below:

1. 12" x 12" Specimens

The equipment used for producing 1 square foot of foamed Fiberfrax® fiber samples is a 2 gallon foam generator consisting of a centrifugal pump, together with a stainless steel beaker, and a recirculating line (See Fig. 2).

A stainless steel beaker with a capacity of 8000 ml was used. At the base of the beaker wall is a coupling outlet which is connected by a circulating line to the centrifugal pump. This line has a ball valve and union used to separate the beaker and pump. Once separated the foam may be discharged from the beaker into the mold.

The centrifugal pump used is a Teel, Bronze close-coupled pump model No. 1P788. The pump motor is a Dayton 1/2 HP motor rated at 3450 RPM. The pump has a 1 inch inlet port with a 3/4 inch outlet port delivering 43 GPM

at a 5 ft. pump head.

The pump outlet is reduced to 1/2 inch and by using a (1/2 inch copper) nipple the circuit is completed using a 3/4 inch Tygon hose to discharge the slurry back into the beaker.

Sample preparation involves the use of a 1 gallon Waring 3-speed blender. The low speed setting (15,500 RPM) was used to effectively separate the fibers. The blending time was 30 seconds and the temperature of water used was 45°C.

The blended mixtures were then poured into the container of the foam generator and surfactant and R-7 resin were added. The foam was generated by circulating the slurry with a pump for 15 minutes. The foam was then poured into a perforated stainless steel mold which allowed the water to drain away. A pair of graphite cloths were used in the bottom and the top of the foam in order to faciliate the removal of the foam from the mold upon completion. The sample was first heat treated to 125°C for 30 minutes. The partially cured foam structure was then pressed to 1" thick. The curing was completed by further heating the foam at 250°C for 45 minutes.

Following is a brief summary of the formulation used for fabricating the 12" x 12" x 1" thick specimen:

Formulation

H_2O	3.9 l
Dispersion Agents:	
long staple fiber	7.5 g
short staple fiber	15.0 g
AAA glass fiber	15.0 g
Surface Active Agent:	
AOS	2.7 ml
Organic Additive:	
Resole R-7	18.0 ml

2. 24" x 30" Specimens

The apparatus used for producing the 24" x 30" samples is basically the same as for 12" x 12" samples except for an increase in capacity. This foam generating system consists of a centrifugal pump, Teel Model 1P798 with a 1.5 HP motor, 95 gpm, 1" ID. piping and a 10 gallon stainless steel pot (see Fig. 3).

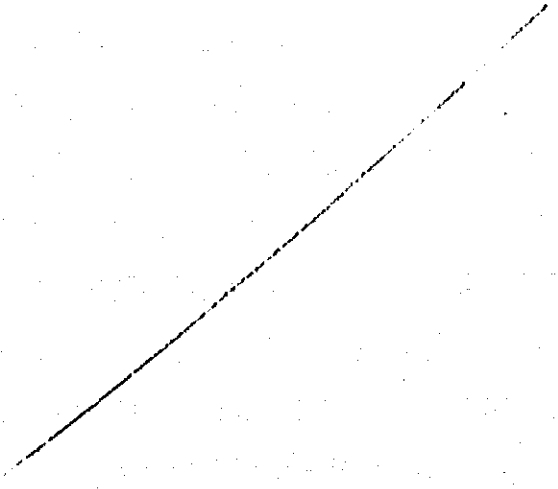
Preparation of the 24" x 30" specimen requires that the slurry components for the fiber break-up step be blended in 8 batches of the same weight ratio as the sample. The blending is done at low speed for 3/4 minutes in the 1 gallon Waring blender. Again the water temperature should be about 45°C to generate a suitable low density foam for pressing.

The sample being restrictive due to its size, is heat-treated at 250°C for 10 minutes prior to pressing. Following the pressing step, the sample is returned to the hot air circulated oven for 1 hour at 250°C to complete the process.

Below is the formulation for the 24" x 30" sample:

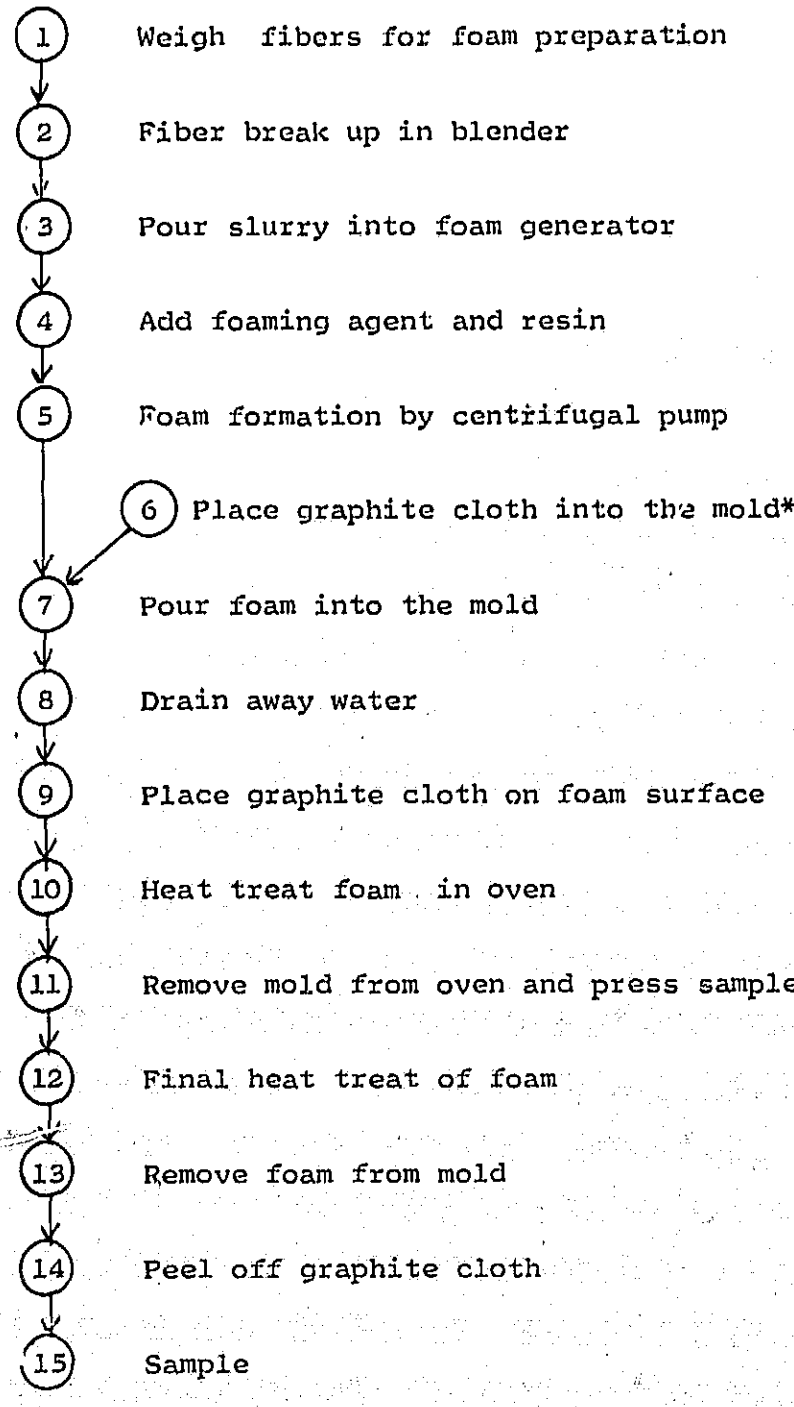
Formulation

H_2O	20.8 l
Dispersion Agents:	
long staple fiber	40.0 g
short staple figer	80.0 g
AAA glass fiber	80.0 g
Surface Active Agent:	
AOS	16.0 ml
Organic Additive:	
Resole R-7	90.0 ml



D. Summary of Fabrication Process

Based on the current modifications the procedures for fabricating the Fiberfrax® can be expressed by the following flow diagram:



* Graphite cloth is used to facilitate the separation of sample from the mold and avoid the formation of resin rich skin on the surface. Same purpose can be served by using glass cloth.

The range of conditions for five key steps is shown below:

(1) Weigh fibers for foam preparation

- Fibers to be dispersed should be blended in amounts such that the batch fiber weight ratio is equivalent to the sample fiber weight ratio.

(2) Fiber break up in blender

- Lab scale - Ronson (Heater/Blender)

  Speed 6 (2550 RPM) 1~3 mins.

- Scale up - Waring 1 Gal. Blender

  Speed Lo (15,500 RPM) 15 secs.~45 secs.

(5) Foam formation by centrifugal pump

- 2 Gal. System - (43 GPM) - 15 mins.~20 mins.
- 10 Gal. System - (95 GPM) - 15 mins.~20 mins.

(10) Heat treat foam in oven

- Temperature ranges

  12" x 12" - 125°C - 30 mins.

  24" x 30" - 250°C - 10 mins.

(12) Final heat treat of foam

- Temperature ranges

  12" x 12" - 250°C - 45 mins.

  24" x 30" - 250°C - 60 mins.~ 90 mins.

It is conceivable that the production of Fiberfrax® foam can be accomplished by either a batch or a continuous operation. The development and optimization of process conditions for manufacturing the Fiberfrax® foam in large quantities will be the major task to be accomplished in the future contract.

F. Characterization of Foam Specimens

The foamed Fiberfrax® samples produced were characterized by a number of measurements or tests such as density measurement, TGA analysis, microscopic examination, wicking test, flame impingement test and acoustical attenuation measurement. The last two tests were carried out at NASA and the rest were conducted in our laboratory.

1. Density and Visual Examination

The density of the foamed specimen was determined by dividing the total weight of the sample by the estimated volume. The visual examination of the sample as produced by the added pressing step shows a more uniform surface texture. The internal areas as exposed when the wicking samples were prepared further exhibited the uniform fiber structure through the cross section.

The following table shows the results of the samples fabricated by the modified process.

Fiberfrax® Blend/AAA Micro-Fiber

Size	Density Range (lbs/cu.ft.)	Average Density (lbs/cu.ft.)
12" x 12"	.77 - .99	.85
24" x 30"	.74 - 1.00	.88

2. Thermogravimetric Analysis

The purpose of this study was to investigate the thermal behavior of the resin bonded foam samples. The samples studied were blends of the following weight ratios as are the final specimens:

	Fiberfrax®			
Sample	HiFi	Short	Long	AAA
A	2		1	2 (post-treated)
B		2	1	2

The TGA was carried out both in air and nitrogen atmosphere up ro 800°C. The thermograms shown in Figures 4 .nd 5 illustrate the effect of the test on both the surface and body of each sample.

The results indicate the following:

a. Sample A contains about 2% resin.

b. Sample B contains about 1% resin.

c. Surface of Sample A contains about 3.5% resin.

d. Surface of Sample B contains about 2% resin.

e. The increased weight loss in nitrogen atmosphere is due to the glass fiber content as observed in the TGA results of AAA glass fiber alone.

3. Microscopic Examination

This study was made to determine the distribution of the component fibers and the resin binder in the sample structure. The examination of the samples was made under the electron microscope. Figures 6 and 7 respectively show the typical structure of formulations A and B as described in the preceeding section. The fiber distribution appears to be homogeneous in both samples. And the low resin content as determined by the TGA study is further confirmed by these photomicrographs.

Previous examinations of un-pressed specimens showed a tendency for resole to spread out in the bonding area which is not readily visible in these low resin content samples. Furthermore, the current pressed samples maintain their structural integrity with a lesser amount of binder being required. This may well be due to the generation of the

low density foam which is then densified by pressing.

4. Wicking Test of Post-Treated Specimens

The wicking test was performed according to procedures provided by NASA-JSC. The test procedure was originally developed by The Boeing Company (Boeing Material Specification BMS 8-48D). The test specifically determines the wicking properties of the material by supporting a sample vertically in water, then measuring the degree of wicking above the water line. Three insulation foam specimens were included in the test: as received, oven aged and leached samples.

The actual wicking of material as received is determined as follows:

a. Cut six 1 inch by 6 inch specimens from the insulation material with the 6 inch length in the direction of the roll. Cut 6 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 0.025 to 0.035, 4 x 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. Pour distilled water at room temperature into the container to a height of one inch.

c. Position the remaining six specimens similar in another container. Pour distilled water into the container to a height of one inch. Maintain the temperature of the water at 120 ± 5°F. Note the

degree of wicking every 24 hours.

Wicking After Oven Againg - Age the insulation material (approximately 8 x 14 inches), in a forced air circulating oven, at 160 ± 5°F for 2 weeks. Test the aged insulation material for wicking as in a, b, and c, for wicking of material as received.

Wicking After Leaching - Leach insulation material (approximately 8 x 14 inches), per Federal Specification CC-T-191b, Method 5830. The insulation material may be held under water by placing it beneath a submerged galvanized wire screen. Air dry thoroughly and test for wicking as in a, b, and c, for wicking of material as received.

Wicking Requirements - Tested materials must not wick to greater than 1/4 inch above the waterline in 168 hours when tested. In addition, precipitates must not form in the water bearing the wicking specimens. Wetting of the submerged portion of the wicking specimens is permissible. Surface wetting is not considered as wicking but cannot be more than one inch above the waterline.

The wicking test was conducted on samples of Fiberfrax®/ AAA glass micro-fiber with a 1:1 weight ratio. To insure the test completion, three duplicate 12" x 12" x 1" thick specimens were fabricated and post-treated with Scotchban® FC-805 paper size. The average temperature for the oven aged sample was 162.6°F and 83.8°F for the leached sample. The wicking test apparatus used and the degree of wicking on samples without post-treatment are shown in Figures 8 and 9 respectively. Table 3 shows the average results of

the above samples.

Post-treatment of the samples is the result of wicking studies carried out in the development contract last year. Without this procedure the degree of wicking exceeded the allowable limit of the test. The post-treatment consists of a 2 step treatment with Scotchban® FC-805 paper size, a water soluble fluorochemical manufactured by 3M Company.

First, the three 12" x 12" specimens were surface treated. This allows the structure to maintain its form while being immersed in a solution of FC805 (10% by sample weight), the second step of the treatment. After each step the sample is cured at 100°C. The surface treating eliminates a problem arising after the leaching of a sample. Upon removal from the water, the weight of the retained water has a collapsing effect on the foam. Samples tested showed losses of approximately 10% in thickness after drying. These treated samples now exhibit a buoyant property requiring a force be exerted on the sample to keep it submerged during leaching.

The results of all three sets of samples show that no wicking occurs other than condensation on the samples supported in the heated water container. Removal of the samples from their respective trays shows that the submerged portions have been completely wetted, but this is allowable.

5. Flame Impingement Test

The flame impingement tests for the Fiberfrax®/Glass fiber blends fabricated were conducted by the Structural

Test Branch at NASA Lyndon B. Johnson Space Center, Houston, Texas. Prior to testing of the full size samples it was determined that previously tested samples this contract period were subjected to conditions more severe than required. The adjustment made was to locate the full size samples at a distance of 5 inches from the flame source rather than 3 inches to simulate the heat flux of JP4 fuel. Results of tests are presented in Figures 10 and 11.

Figure 10 presents an earlier comparison of the backface temperature of Fiberfrax® foamed insulation with a density of 0.8 lbs/cu.ft. with those of other competitive insulators. The result for the Fiberfrax® blend was superior to the other materials of the same density level.

In Figure 11 the results for two sets of full size (24" x 30") Fiberfrax® glass fiber blends are shown. Both of these formulations were stated above in the discussion of Thermogravimetric Analysis. Each of these samples satisfactorily survived through the test period of 10 minutes. The AAA glass fiber content, reduced to 40% in these samples, serves a two-fold purpose. Initially its use was for improvement of acoustical attenuation due to its fine diameter. But it also serves to act as a binder at the higher temperatures where it melts and replaces the organic resin which burns off earlier during the test. Of the two formulations, those pieces comprising sample set "B" produce the lowest backface temperature.

6. Acoustical Test

The acoustical test of all the Fiberfrax® samples was also conducted at NASA Lyndon B. Johnson Space Center. The "Quick-Look Report--Activation of Aircraft Insulation Acoustic Test Apparatus" was used as the reference for the test. The noise reduction of the insulation foam was measured according to specification BMS 8-48D (Boeing).

During the contract period a number of samples, both 12" x 12" and 24" x 30", were submitted to NASA for testing. The complete results are tabulated in Table 4.

The test results show that the two formulations of the Fiberfrax®/AAA glass micro-fiber blend supplied as the final full size specimens both have achieved the acoustic requirements for the insulation material. Again, these formulations are described above in the Thermogravimetric Analysis section. Here again sample "B", using Short Staple Fibers in place of HiFi fibers, has resulted in slightly better noise reduction values than the sample "A" formulation.

TABLE 1

Fiberfrax® Properties

Fiber Type	Composition Al_2O_3 %	Composition SiO_2 %	Fiber Diameter (Mean)	Fiber Length in.	Temperature Continuous Use °F	Melt Point °F	Density gms/cc
H-Bulk	62.0	38.0	2-4	to 1	2600	3500	2.60
Bulk (short staple)	51.7	47.6	2-3	to 4	2300	3260	2.53
Chopped	51.7	47.6	2-3	300	2300	3260	2.53
Milled	51.7	47.6	2-3	14	2300	3260	2.53
Hi-Fi	51.7	47.6	1.6	0.5	2300	3200	
Long Staple Fine	43.9	50.1	8	to 10	2300	3260	2.62

TABLE 2

Surface Active Agents

NAME	TYPE
Methyl-p-toluene Sulfonate	Alkyl, Aryl Sulfonate
Liquofoam[1]	
Sulframin 1260 Slurry[2]	Alkyl Aryl Sulfonate
Sulframin AOS Slurry[2]	Alpha Olefin Sulfate
Ultra Sulfate SL-1[2]	Sodium Lauryl Sulfate
Ultra Sulfate SE-5[2]	Alcohol Ether Sulfate
Triton X-151[3]	Alkyl Aryl Sulfonate
Emulsifier AH-861[3]	Alkyl Aryl Sulfonate

1 - Mearl Corp.

2 - Witco Chemical Corp.

3 - Rohm and Haas

TABLE 3

Wicking Test Results

Showing average amount of condensation above water line

Sample Condition	Avg. Temp. (°F)	Sample Taken From Length (in.)			Sample Taken From Width (in.)		
As received	67.1	0.000	0.000	0.000	0.000	0.000	0.000
920	119.9	0.000	0.214	0.205	0.205	0.214	0.214
Oven aged	73.7	0.000	0.000	0.000	0.000	0.000	0.000
922	120.7	0.313	0.313	0.313	0.271	0.271	0.271
Leached	75.0	0.000	0.000	0.000	0.000	0.000	0.000
921	121.7	0.313	0.259	0.250	0.250	0.250	0.250

All the above samples were post-treated with Scotchban® FC-805 paper sizing.

TABLE 4

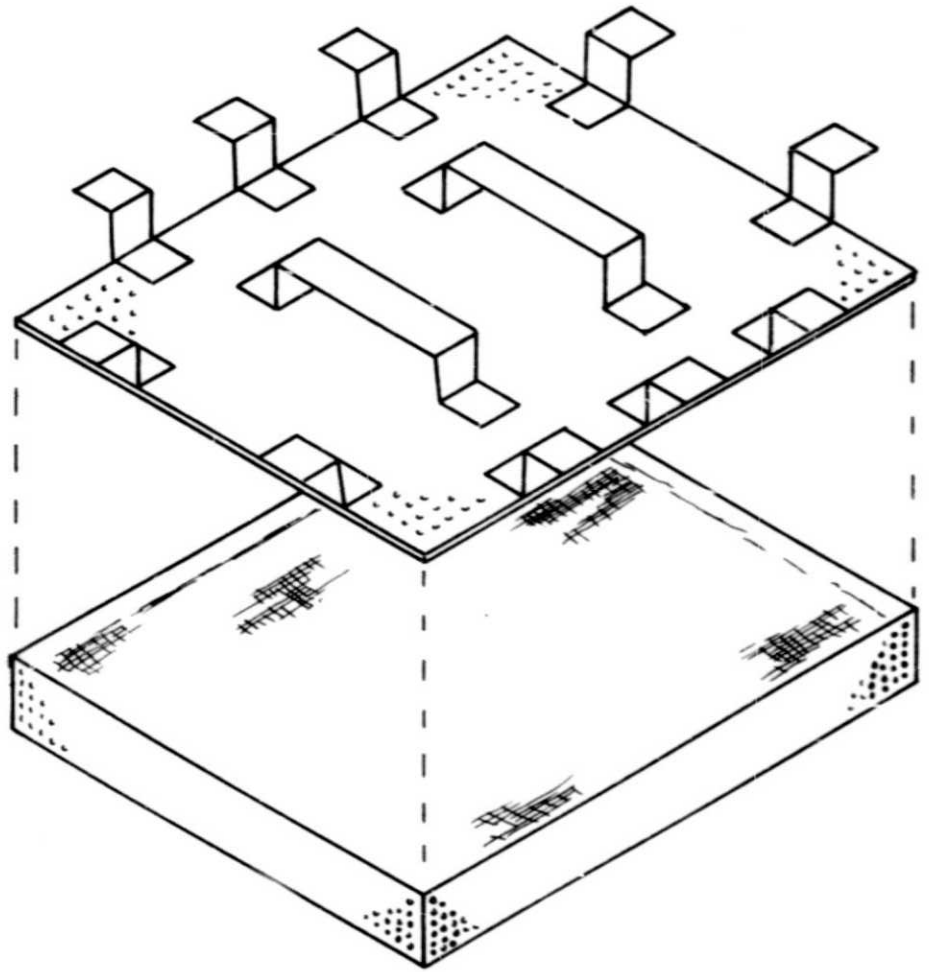
Acoustic Attenuation Comparison of Standard Aircraft Fiberglass Insulation and Fiberfrax®/Glass Fiber Foam Blends

Material	Sample Composition and Glass Content	Thickness Inches	Density lbs/ft^3	Noise Reduction, dB 1000 Hz	2000 Hz	4000 Hz
Requirements (26" x 26")		3	0.60	8.0	19.0	27.0
Fiberglass (12" x 12")		3	0.60	7.8	17.0	22.2
Fiberfrax® Foams (12" x 12")						
Sample 795	LS/SSW/AA-33-1/3%	1	0.81	1.5	4.0	5.0
" 795, 802	"	2	0.86	3.5	7.0	9.5
" 795, 802, 809	"	3	0.85	5.0	10.0	13.0
" 812	LS/AA-50%	1	0.85	2.0	5.5	7.5
" 812, 815	"	2	0.84	4.0	9.5	13.0
" 812, 815, 816	"	3	0.84	6.5	12.5	18.5
" 900	LS/AA-50%	1	0.87	2.0	5.5	6.5
" 900, 910	"	2	0.84	4.5	9.5	12.5
" 900, 910, 915	"	3	0.88	7.5	14.0	18.5
" 904	HF/AAA-50%	1	0.85	3.0	7.0	9.5
" 904, 907	"	2	0.86	6.5	12.5	17.5
" 904, 907, 911	"	3	0.87	9.0	17.0	22.5
" 912	LS/HF/AAA-40%	1	0.90	3.5	5.0	10.0
" 912, 913	"	2	0.89	7.0	9.0	18.0
" 912, 913, 914	"	3	0.87	9.5	16.5	24.0
" 997, 999, 1001	LS/HF/AAA-40%	3	0.85	7.5	13.8	18.0
" 1002, 1003, 1005	LS/HF/AAA-40%	3	0.89	8.0	15.7	19.5

TABLE 4 Cont'd.

Acoustic Attenuation Comparison of Standard Aircraft Fiberglass Insulation and Fiberfrax®/Glass Fiber Foam Blends

Material	Sample Composition and Glass Content	Thickness Inches	Density lbs/ft^3	Noise Reduction, dB 1000 Hz	2000 Hz	4000 Hz
Fiberfrax® Foams (24" x 30")						
Sample 961	LF/HF/AAA-40%	1	0.93	4.5	7.5	11.0
" 961, 969	"	2	0.93	9.8	13.0	19.0
" 961, 969, 970	"	3	0.91	11.0	19.5	26.0
" 989	LS/SSW/AAA-40%	1	0.88	6.3	8.8	12.3
" 989, 992	"	2	0.89	11.0	14.8	20.6
" 989, 992, 993	"	3	0.88	13.0	20.8	26.8



Plunger - Tempered Masonite (3/8" thick with 1/4" holes, with pre-set stops

Mold - Perforated Stainless Steel (24" x 20" x 4" deep) containing low density foam between two pieces of graphite cloth

Figure 1 - Foam Pressing Method

Figure 2 - Two Gallon Foam Generator

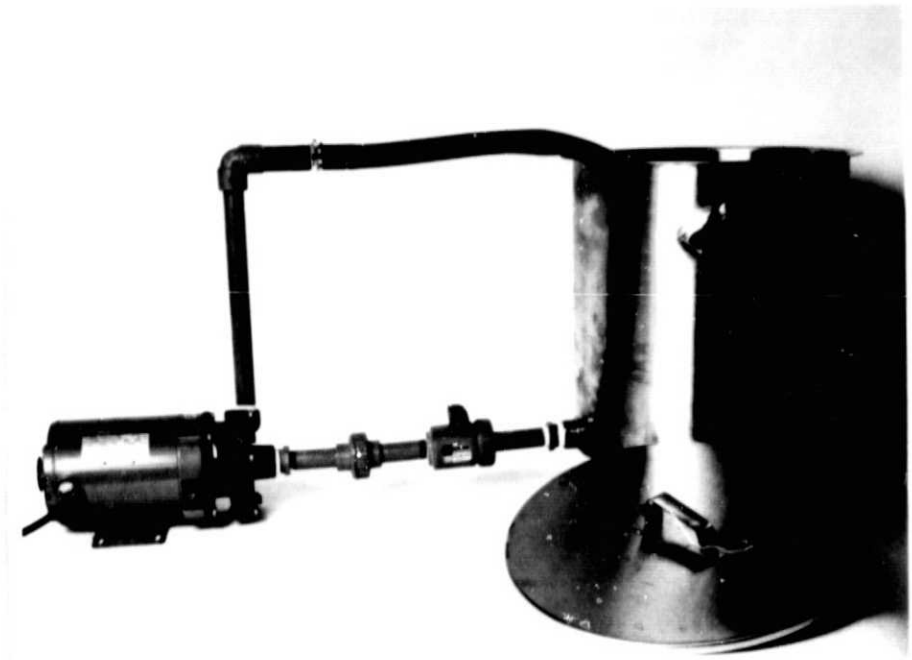


Figure 3 - Ten Gallon Foam Generator

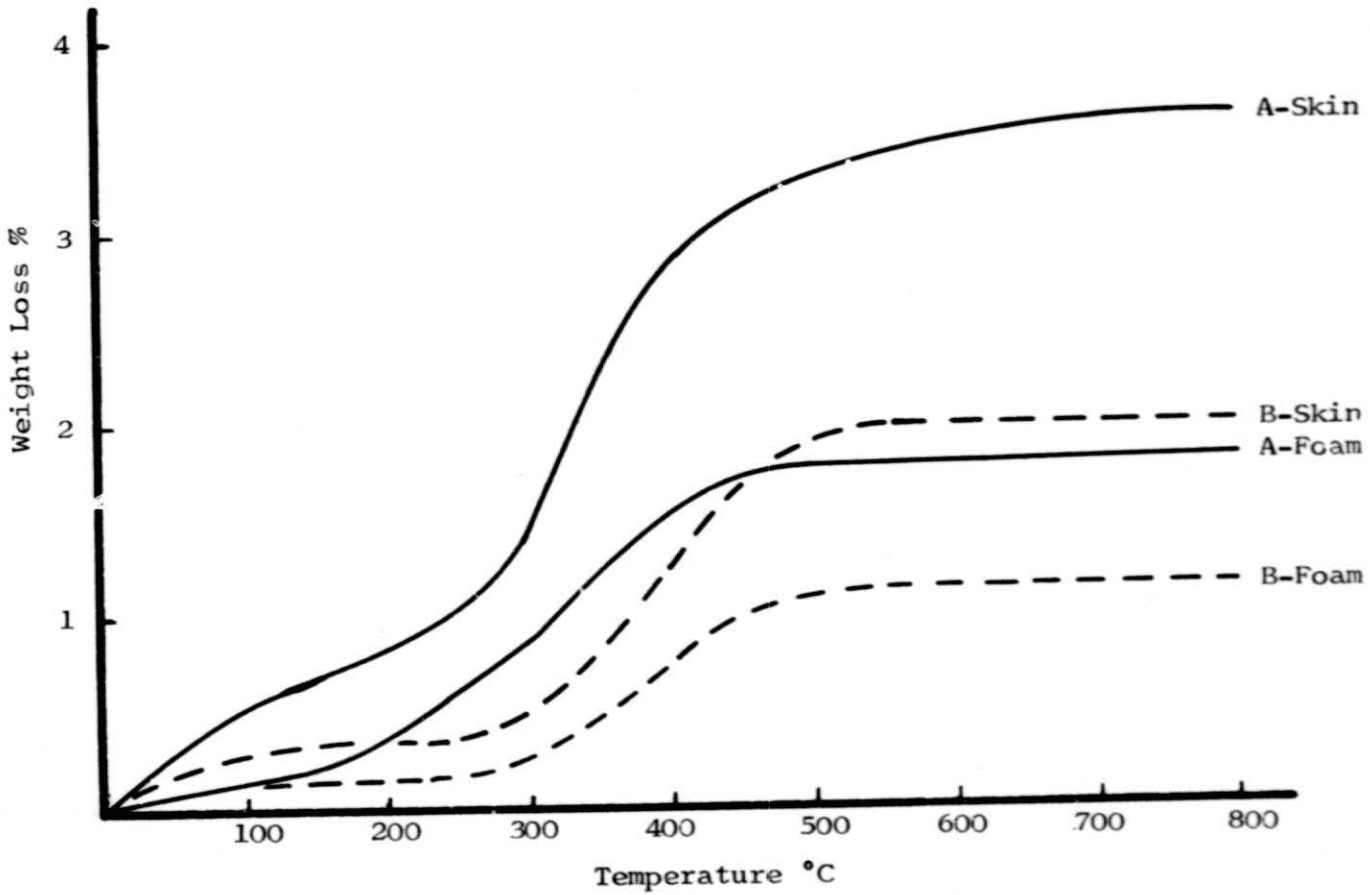


Figure 4 - TGA of Fiberfrax®/AAA Foam Samples in Air

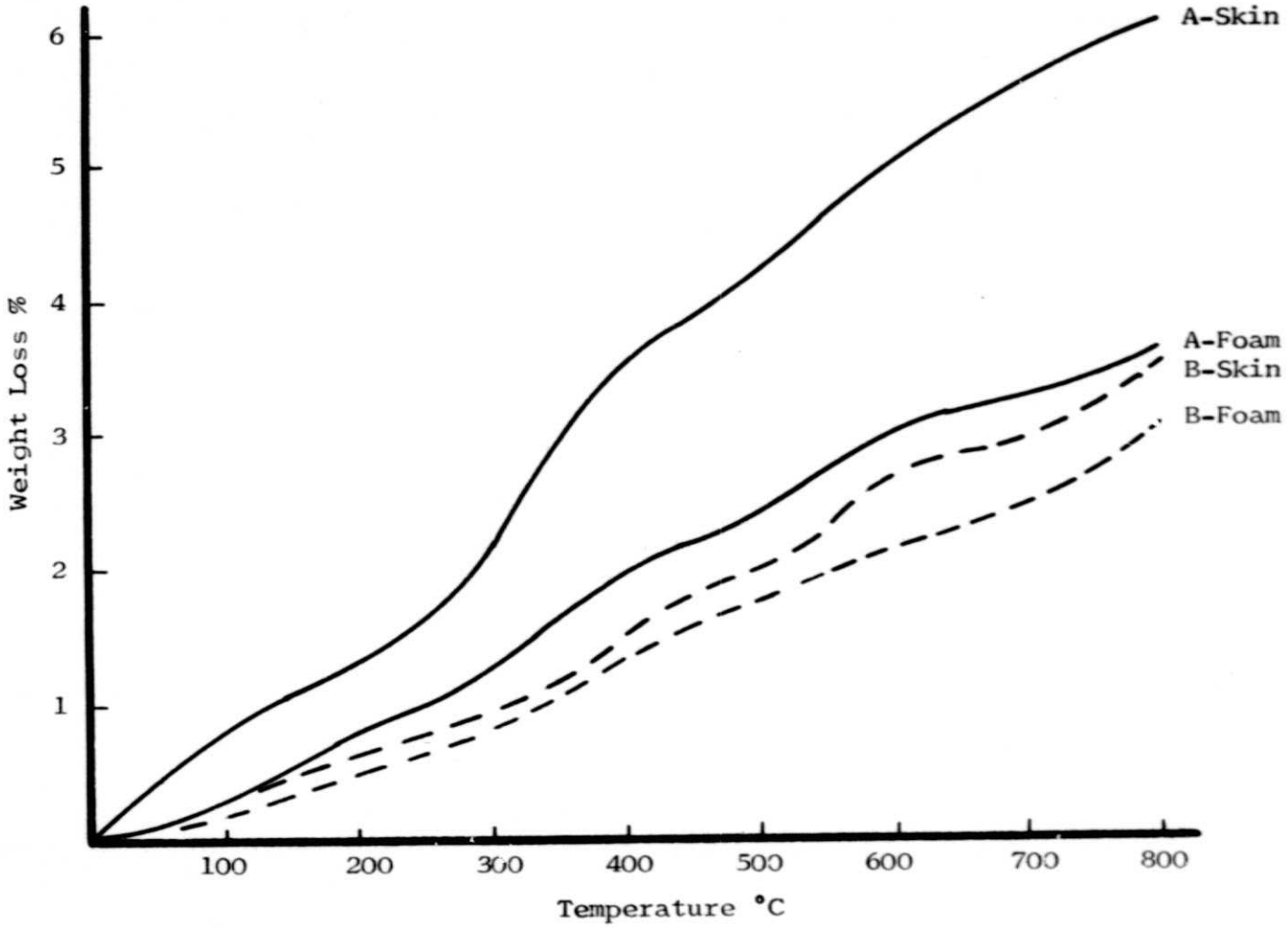


Figure 5 - TGA of Fiberfrax®/AAA Foam Samples in Nitrogen Atmosphere

Figure 6 - Electron Photomicrograph of Fiberfrax®

Formulation A

Sample Composition

15.0 g	AAA Glass Fiber	
7.5 g	Long Staple Fiber)	Fiberfrax®
15.0 g	HiFi Fiber)	
2.7 ml	Sulframin® AOS Slurry	
18.0 ml	Resole R-7	
3.9 l	Water	

Figure 7 - Electron Photomicrograph of Fiberfrax®

Formulation B

Sample Composition

15.0 g	AAA Glass Fiber	
7.5 g	Long Staple Fiber	Fiberfrax®
15.0 g	HiFiFiber	Fiberfrax®
2.7 ml	Sulframin® AOS Slurry	
18.0 ml	Resole R-7	
3.9 l	Water	

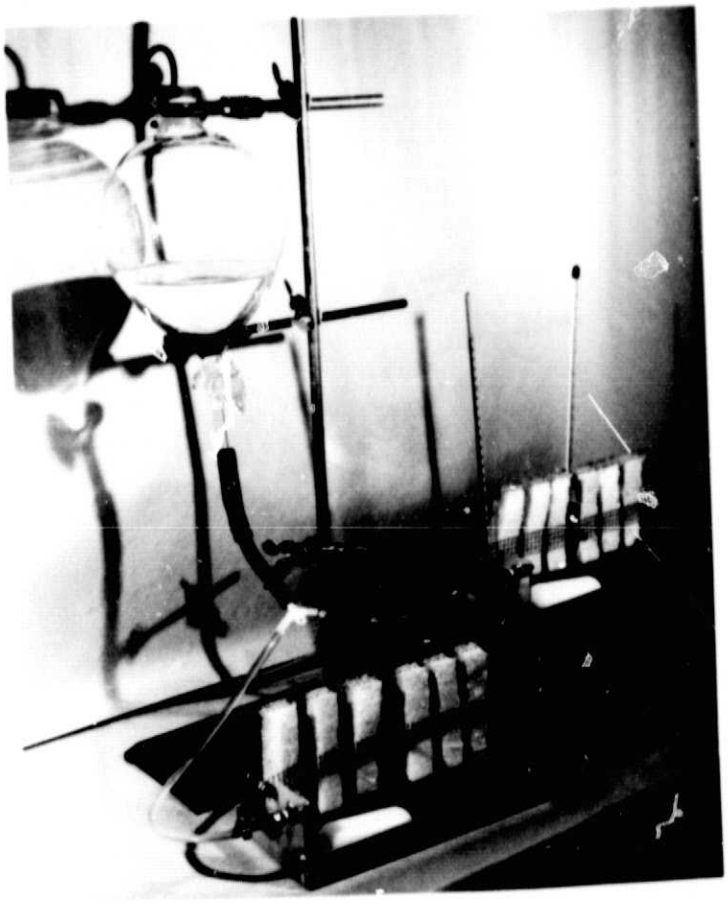


Figure 8 - Laboratory Set-up for Wicking Test

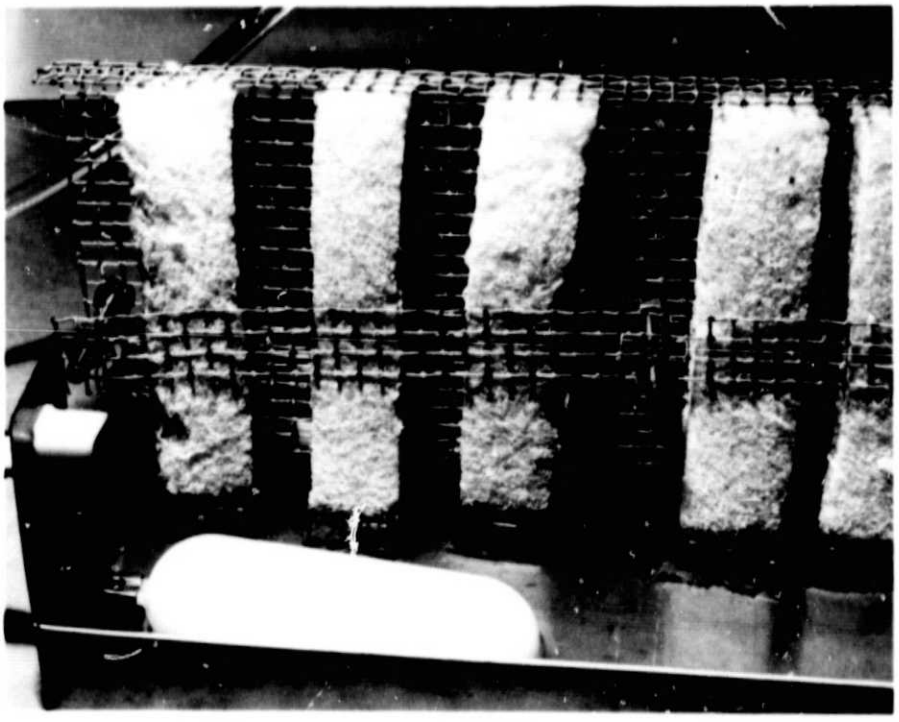


Figure 9 - Degree of Wicking of Fiberfrax® Foams

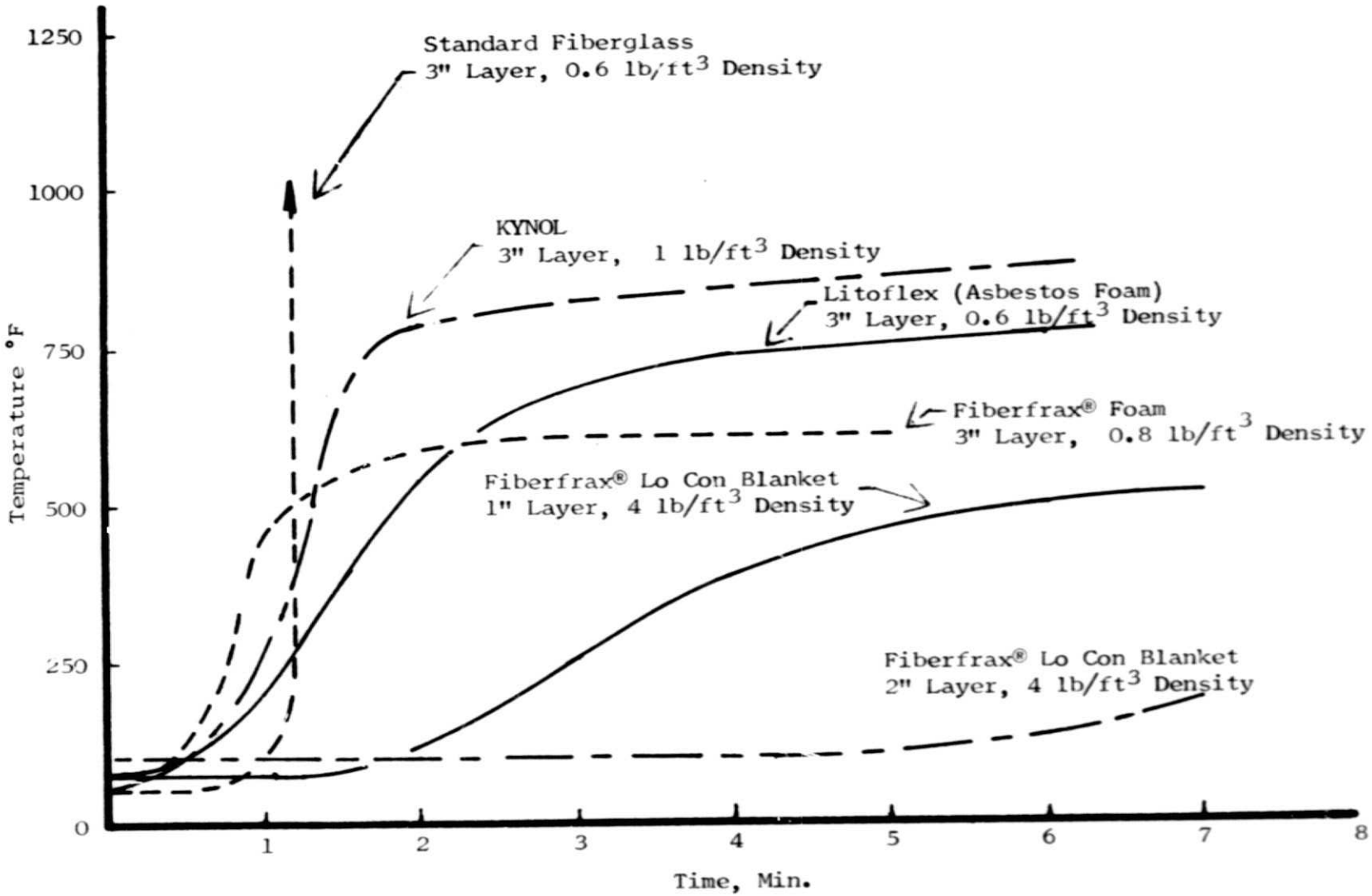


Figure 10 - Flame Impingement Tests for Various Insulators

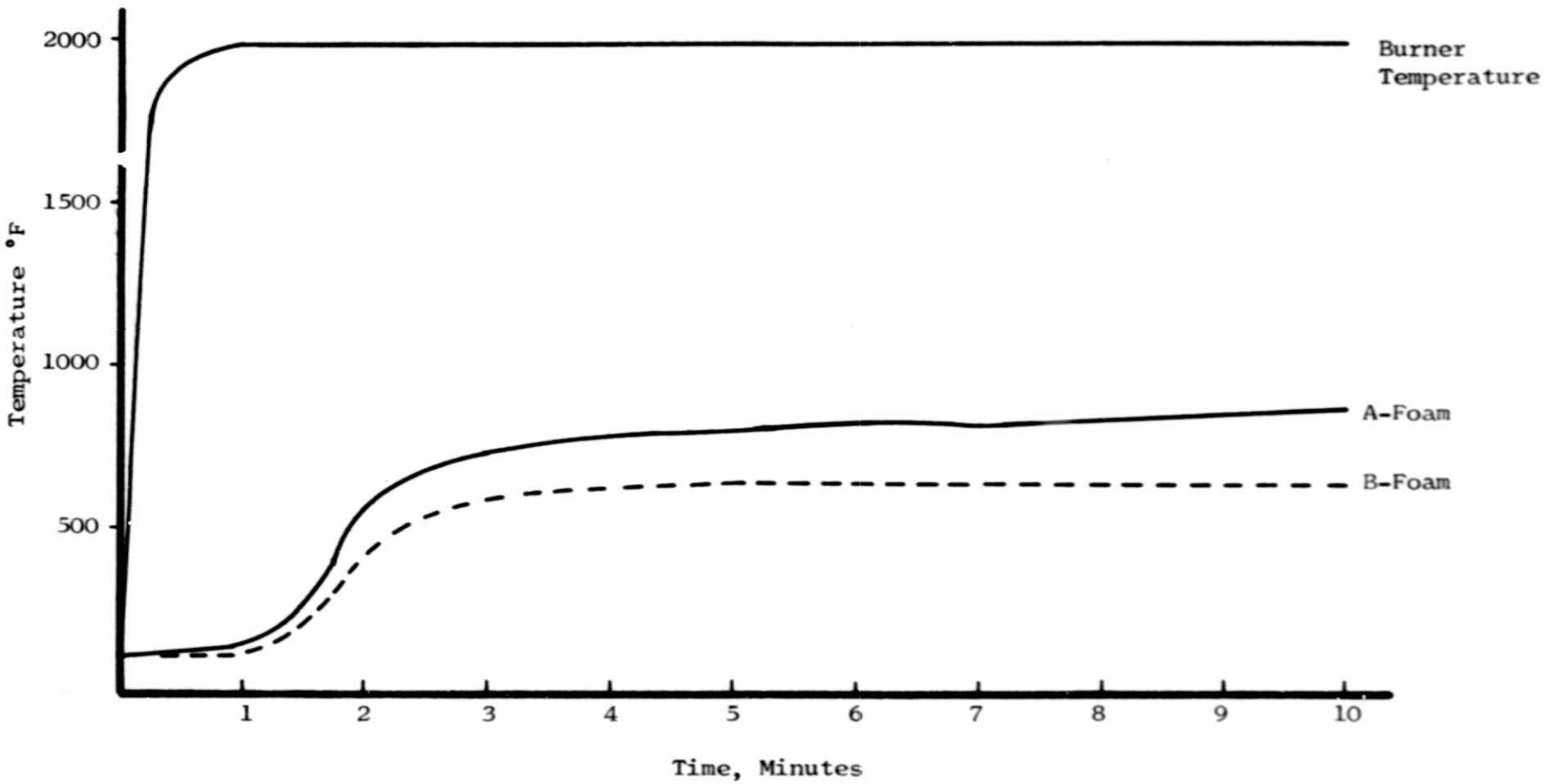


Figure 11 - Flame Impingement Test for Full Size (24" x 30")
Fiberfrax®/Glass Fiber Blends