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DEVELOPMENT OF A THERMAL ACOUSTICAL AIRCRAFT INSULATION MATERIAL

Ruey Y. Lin
Edward A. Struzik

Final Technical Report

PRICES SUBJECT TO CHANGE

Prepared Under Contract NAS 9-13641
November 1974

Research and Development Division, Niagara Falls, New York 14302

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I. INTRODUCTION

In spite of ever-increasing awareness of improvement of aircraft safety there are still many critical areas needed to be resolved. In recent years, particular attention has been focused on the increase of safety margin against fire caused by accident. Under the NASA Aircraft Flammability Program substantial efforts have been concentrated in the following areas:

A. Improve cabin materials/electrical components.

B. Develop thermal resistant window.

C. Improve fuselage insulation--thermal and acoustical.

This report deals with the development efforts in the third area.

Currently resin bonded fiberglass battings are widely used in the aircraft primarily because of its excellent acoustical behaviour. The major shortcoming of this insulator is that it provides no thermal protection owing to its poor flame impingement properties. A light weight asbestos foam material was developed recently (U.S. Patent 3,338,994) and was once considered as a prime candidate material to replace the fiberglass insulator. It was ruled out for such an application because of the health hazard associated with the asbestos.

Starting October 1, 1973 a program on "Development of a Thermal/Acoustical Aircraft Insulation Material" was initiated in the Research and Development Division of The Carborundum Company under a contract sponsored by NASA, NAS 9-13641. The objective of the program was to develop a process for fabricating light weight foams, primarily based on Fiberfrax®, suitable for thermal and acoustical insulation in aircrafts.

The process described in U.S. Patent 3,338,994 was used as the initial reference for fabricating Fiberfrax® foams. It was found, however, that the process was not applicable to the Fiberfrax® system. Through the modification of process procedure, careful screening of foaming agents, and addition of proper resin to the system, a light weight thermal/acoustical Fiberfrax® foam was finally developed.

In the subsequent sections discussions will be devoted to process development, material screening, process optimization and product characterization.

II. SUMMARY OF RESULTS

During the contract period covering October 1, 1973 through June 30, 1974 our efforts were focused in a feasibility study with the objective to fabricate a low density foam structure meeting the criteria of requirement. Specimens produced in the contract period were characterized both in Carborundum and NASA for physical, thermal, and acoustical properties. Important accomplishments are summarized below:

A. The procedures and apparatus for fabricating Fiberfrax® foams were established. In the area of process development, the fabrication went through a series of scale-up operations from a laboratory scale sample of 4" x 4" to a final test specimen of 24" x 30".

B. The process for fabricating Fiberfrax® foam was successfully extended to KYNOL and Fiberfrax®-KYNOL mixed systems.

C. Foam specimens with a density in the range of 0.6 to 1.2 lbs/cu.ft. were consistently prepared and the techniques to vary the density were also developed.

D. Two resin systems, resole (R-7) and Alpha Resitron® resin from Ventron Corporation were found to be the effective organic binders for the foams.

E. Flame impingement tests carried out at NASA showed that the Fiberfrax® foam displayed excellent thermal properties.

F. The acoustical attenuation of the foam specimen was still not up to the desirable level. The addition of fine diameter glass fiber, Grade AAA from Johns-Manville, to the system effectively improved the acoustics of the foam.

III. FUTURE WORK

The work carried out thus far has clearly demonstrated the feasibility of fabricating a new light weight foam structure based on Fiberfrax® and other dispersing fibers. It is important to capitalize this technology by further optimizing the system and developing it into a stage ready for manufacturing. Immediate results from the continuation of this program will be the production of sufficient Fiberfrax® foam for the external fuel-fire test at NASA which is scheduled in 1975. The long range effect of this development will be the availability of a new thermal/acoustical insulation material to improve the safety of future aircrafts. It is proposed that the future efforts should be devoted in the following areas:

A. Further improvement of acoustical attenuation property and mechanical strength of the foam. These will be achieved by adding the fine diameter glass fiber in the system and by evaluating new organic resin as the binder.

B. Development of a production process for manufacturing the Fiberfrax® foam. In this phase of work the objective will be to develop an efficient process for making the Fiberfrax® foam in larger quantity economically and consistently. The reliable quality control guideline will also be established.

C. Fabrication of sufficient quantity of Fiberfrax® foam for an external fuel-fire test on a fuselage at NASA. The data from this test will be a significant measure for the effectiveness of the foam as an insulation against fire.

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(s) 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. The conditions used to cure the specimens depends mainly upon the type of resin used in

the system.

It was with this general process that a low density Fiberfrax® structure was developed. In the initial bimonthly contract period our effort was focused on establishing the basic foaming procedures. All the specimens (4" x 4") were fabricated on a laboratory scale. Once the feasibility was demonstrated the emphasis was shifted to making larger specimens in a sequential order: first medium size, 8" x 8" and 12" x 12", and finally full size 24" x 30". It should be mentioned that a modification of process conditions was usually required as the size of the specimens increased from one level to another. During the entire contract period close to 800 specimens were prepared. The fabrication conditions for part of the runs were summarized in the bimonthly progress reports. The properties of the Fiberfrax® foams were affected by the material parameters such as fiber length and diameter, surface active agent (foaming agent), and organic resin used in the system. The importance of these material parameters is discussed in the next section.

B. Optimization of Important Material Parameters

1. Fiberfrax®

The dispersion fiber used in producing a light weight thermal/acoustical aircraft insulation material is a ceramic fiber, Fiberfrax®. Several grades of Fiberfrax® fiber are available, and they provide the possibility of selecting the proper fiber characteristics for controlling the foam products. Those grades of fibers slected for evaluation are: H-bulk, Bulk (short staple and short staple washed),

chopped, milled, Hi-Fi, and long staple fine. Table 1 lists the properties of the above named fibers. All these fibers offer very good insulating properties at 2300°F for continuous usage.

The Hi-Fi grade Fiberfrax® fiber was used in the initial attempt in the foam development. Because of the favorable physical sizes, 0.5 inches in average length and 1.6 μ in average diameter, this fiber dispersed quite uniformly in the foaming action. The resulting foam appeared to be lack of strength due to insufficient mechanical interlocking of fibers.

The effort was then focused on evaluating other Fiberfrax® fibers as a replacement for Hi-Fi. Samples from bulk short staple fiber possessed a dense mat appearance. Specimens from the milled and chopped fiber were relatively dense and void free. No appreciable increase of strength was noticed. On the other hand, when the long staple fine fiber was used the strength of the foam was greatly improved. However, the structure of the foam was too open to be desirable as an insulation material. Thus, it was concluded that any single Fiberfrax® fiber was not suitable for foam fabrication purposes. After extensive investigation the Fiberfrax® foams with satisfactory structure and strength were fabricated by proper blending of various fibers.

2. Surface Active Agents

Surface active agents were used to facilitate wetting and foam formation of the Fiberfrax®/water slurry. The surface active agents evaluated in this study included:

methyl-p-toluene sulfonate, liquifoam, Ultra® sulfate SE-5, Ultra® sulfate SL-1, Sulframin® AOS slurry, Sulframin® 1260 slurry, Triton® X-151 and Emulsifier AH-861. The trade name, chemical nature and manufacturer of these agents are summarized in Table 2. All these surfactants were investigated in our initial screening study. The optimum amount of surface active agent needed depends upon the type of surfactant used and also the mechanical means by which the foam is formed. As a rule the more the surfactant used the lower the foam density. To determine the effectiveness of various surfactants the quantity of surfactant to produce a same foam height, say 1", was defined. Results for 4" x 4", 12" x 12" and 24" x 30" specimens are summarized below:

	4" x 4"*1	12" x 12"*2	24" x 30"*3
Emulsifier AH-861	1.00 ml	27 ml	337.5 ml
Ultra® Sulfate SE-5	0.14	3.6	-
Sulframin® AOS Slurry	0.30	2.7	10 ml
Sulframin® 1260 Slurry	0.14	3.6	-
Ultra® Sulfate SL-1	0.50	2.50	-
Triton® X-151	1.00	27.0	-
Liquifoam	0.27	35.1	-
Methyl-p-toluene sulfate	0.27 gm	2.43 gm	-
Sodium lauryl sulfate	0.6 gm	0.6 gm	-

Notes: *1 Foaming was carried out in a high speed blend

*2 Foaming was performed in a 2 gallon foam generator

*3 Foaming was conducted in a 10 gallon foam generator

It should be pointed out that the foam structure depends,

to a great extent, on the surfactant used. In our study most specimens were produced with AH-861, Sulframin AOS and Ultra Sulfate SE-5.

C. Resin Binder

Attempts to produce a flexible lightweight insulating material by using a water soluble soap and bivalent metal salt as binding agent was initially investigated. The mechanical strength of the resulting foam was too weak to be desirable. It was found that addition of a small amount of organic resin as binder was necessary. Two high temperature organic resins were evaluated, R-7 (a resole) and Resitron® Alpha Resin from Ventron Corporation. Both of these two resins are water soluble and are very easy to incorporate them in the foaming process.

1. R-7 Resin

During most of the contract period, the effort was placed on developing a suitable insulation using R-7 as the resin bonding agent. Various combinations of resin and surfactant amounts were tried together with curing conditions to develop a working composition. Dependent on sample size, as previously noted in discussion on surfactants, the resin required was determined together with the appropriate curing conditions. Changes in one of these parameters dictated an adjustment in one or more of the remaining parameters to produce a homogeneous, well bonded material.

Summarized in the following list are the amounts of R-7 resin and the curing conditions for the respective samples.

Size	Amount	Curing Time	- Temperature
4" x 4"	1.0 ml	0.5 hr.	250°C
8" x 8"	8.0 ml	0.5 hr.	250°C
12" x 12"	18.0 ml	0.5 hr.	250°C
24" x 30"	157.5 ml	1.0 hr.	250°C

2. Resitron® Alpha Resin

Resitron® alpha resin like R-7 is a water soluble resin system and lends itself easily to the Fiberfrax® foam process. Resitron® is manufactured by Ventron Corporation of Beverly, Massachusetts and is supplied as a two component system.

In addition to the proper amounts of resin required for the sample, the ratio of former to hardener had to be determined as well. Three former/hardener ratios were tried: 1/1, 2/1, 3/1. The second combination, 2 parts former and 1 part hardener consistently provided the best sample.

The best foaming agent for the Resitron® resin bonded sample was found to be AOS slurry. The temperature required for curing was much lower than that for R-7 resin, 150°C.

Below are the amounts of Resitron® resin (at a ratio of 2/1) and the curing condition used for various specimen sizes.

Size	Amount	Curing Time	- Temperature
4" x 4"	3.0 gm.	0.5 hr.	150°C
12" x 12"	67.5 gm.	1.0 hr.	150°C
24" x 30"	270 gm.	2.0 hr.	150°C

D. Fabrication of Fiberfrax® Foam Specimens

The fabrication of final 24" x 30" test specimens of Fiberfrax® foam was developed by a systematic approach:

- 4" x 4" specimens: establish the basic procedures in a laboratory operation.
- 12" x 12" specimens: scale up the operation by modifying some process conditions.
- 24" x 30" specimens" fabrication of final test specimens based on the technique developed for 12" x 12" samples.

Procedures and results of each stage of operation are summarized below:

1. 4" x 4" Specimens

To 6.0 grams of Fiberfrax® fibers (the standard amount for either one grade or a blend of fibers) 220 ml of deionized water is added and blended at speed 6 (2550 RPM) for one minute. Next, 1.0 ml (quantity determined by surfactant used) of surface active agent is added and blended at speed 10 (3500 RPM) for one minute. Following this, 1.0 ml of Resole R-7 is added with an additional 1/2 minute of blending time. The foamed sample is then poured into the 4" x 4" x 3" deep screen mold, excess water allowed to drain and then placed into the air circulated drying oven at 250°C for 30 minutes producing a well bonded homogeneous specimen.

Standard formulation for the 4" x 4" foam sample was:

H_2O: 220 ml

Dispersion Agents:

long staple fiber: 4 g

short staple fiber: 2.0 g

Surface Active Agents

AH-861: 1 ml

Organic Additive:

Resole (R-7) 1 ml

2. 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. 1).

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.

Samples were prepared using 9 times the standard formulation (4" x 4") in the 2 gallon capacity set-up to produce the 12" x 12" foam.

Reduction in sample preparation time was achieved through the use of a 1 gallon Waring 3 speed blendor in the fiber break-up step. The larger capacity and higher speed, 15,500 RPM vs. 2550 RPM, allowed the blending of the total 9 batch sample. This reduced the blending step from 1 minute per sample to 15 seconds for the batch, which in turn produced a more homogeneous structure.

Initially, the foam formation time using the centrifugal pump was 1 hour. This has been reduced to a circulation time of 15 minutes producing a cream like foam 2 to 3 times the original volume. The foam is then poured into a mold made of perforated sheet metal that allows excess water drainage. Following this the foam is heat treated at 250°C for 30 minutes in an oven to complete the process.

Following is a brief summary of the formulation and process currently used for fabricating the 12" x 12" specimens.

Formulation

H_2O:	2 l
Dispersion Agents:	
long staple fiber:	36.0 g
short staple fiber:	18.0 g
Surface Active Agent:	
AH-861:	27 ml
Organic Additive:	
Resole (R-7):	18 ml

3. 24" x 30" Specimens

Due to the increased amount of slurry needed a larger foam generator was assembled to fabricate the 24" x 30" specimens. The apparatus was basically the same as that for making 12" x 12" samples except the increase in pump size and foam container were required. The apparatus consists of a centrifugal pump, Teel Model 1P798 with a 1.5 HP motor, 95 gpm, 1" I.D. piping and a 10 gallon stainless steel pot (See Fig. 2).

Conditions unchanged are the fiber break-up step, foam generation time, and quantity of water used. For the larger sample, the fiber break-up step is repeated 5 times to prepare the dispersion. Each time, an amount equivalent to a 12" x 12" sample is dispersed by blending for 15 seconds and added to the 10 gallon container. Upon completion of the break-up step the surface active agent and organic binder is added. The slurry is then pumped for 15 minutes to generate the foam.

This foam is then poured into the perforated sheet stock mold and allowed to drain. Normally, the excess water is drained in about 5 minutes leaving a firm, cream like foam. Following this the mold is placed in the hot air circulated oven at 250°C for 1 hour to complete the process.

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

H_2O:	10.0 l
Dispersion Agents:	
Long staple fine	180.0 g
Short staple washed	90.0 g
Surface Active Agent:	
AH-861	337.5 ml
Organic Additive:	
Resole (R-7)	135.0 ml

E. Work on KYNOL, KYNOL Fiberfrax® and BN Foams

Experiments were conducted using KYNOL fiber and boron nitride fiber as dispersing phase replacing Fiberfrax® fibers in the foam structure. The purpose was to see if the foaming procedures developed for Fiberfrax® are applicable to other fiber systems. The process for making 4" x 4" samples was employed for evaluating the new fibers.

1. KYNOL

Specimens of KYNOL foam had low densities in the range of 0.56 to 1.18 lbs/cu.ft. They also exhibited good strength properties while being less resilient and non homogeneous in structure. Cutting the KYNOL foam in half shows the fibers in a swirled structure unlike the homogeneous, random fiber structure of Fiberfrax® foam.

2. KYNOL-Fiberfrax®

The good strength properties shown by the KYNOL foam specimens due to better wetting with the organic additive led to blending of Fiberfrax® and KYNOL.

Blends of the two dispersion agents by the formulation as described in C-2 were made using 8% and 16% by specimen weight KYNOL fiber 1/2" long in place of long staple Fiberfrax®. These specimens resulted in respective densities of 1.20 and 0.82 lbs/cu.ft. The samples were resilient, well bonded, and homogeneous in structure.

3. Boron nitride

Boron nitride fiber produced a low density, 0.49 lbs/cu.ft., foam structure such that it was non-homogeneous, exhibited low strength, and lumpy in appearance. The samples had a very uneven, crusty skin rich in organic additive. Whereas beneath the skin there was very little evidence of binder in the fibers.

To improve the sample strength the BN fiber was pre-heated by soaking in a 1% solution of Ludox (colloidal silica - 40% solids). Use of these treated fibers to produce foam specimens showed no improvement over untreated fibers.

F. Fiberfrax® Foam Containing Johns-Manville AAA Glass Fiber

During the later part of the contract period a number of foam specimens containing Johns-Manville Micro-Fiber Grade AAA were fabricated. The addition of the fine diameter (0.5-0.75μ) to the system aimed at improving acoustic attenuation of the foam specimen especially at high frequency level, 4000 Hz. It will be discussed in the latter sections of this report that such a modification did indeed improve the acoustic behaviour of the sample. The fabrication of AAA glass fiber modified foam

specimen was similar to that of standard foams described previously. In our study, both R-7 and Resitron® Alpha resin were used as the binder. Generally speaking, the Resitron® bonded samples appeared to be stronger and more resilient. The typical formulation for 24" x 30" specimens are summarized below:

1. R-7 Bonded Samples

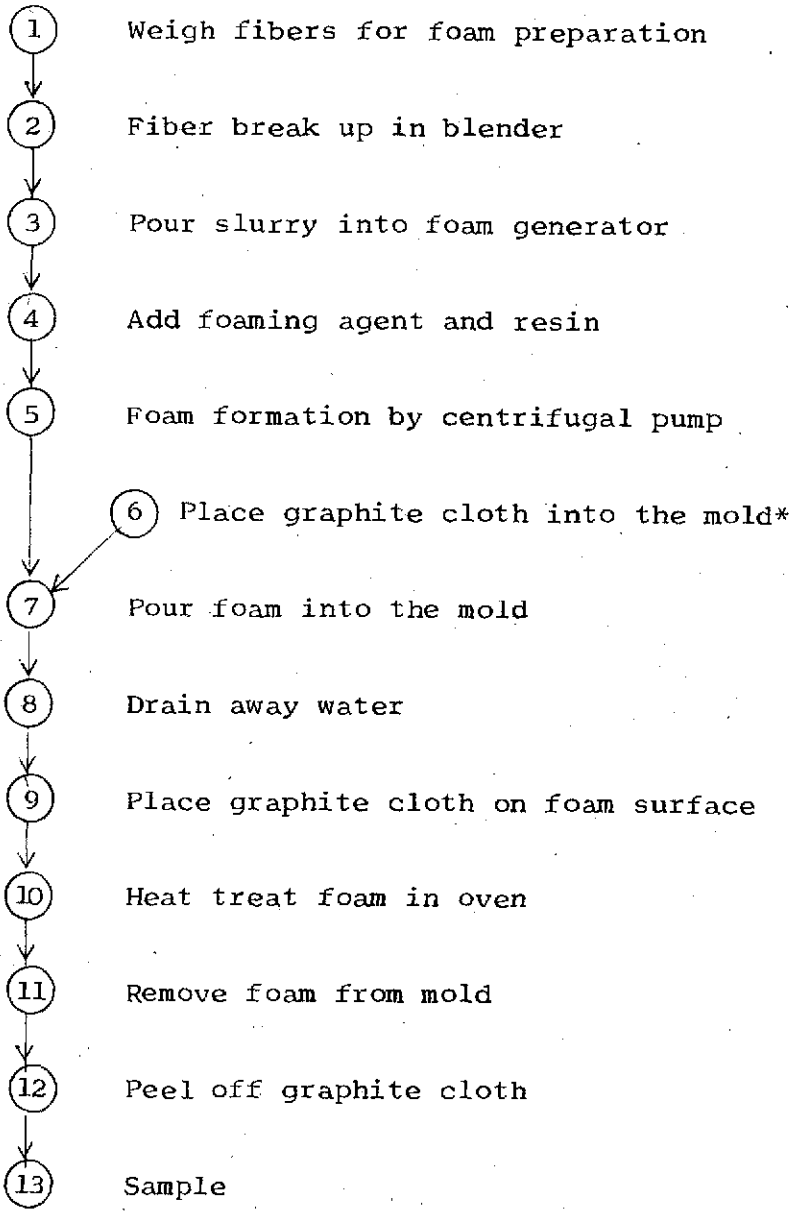
H_2O:	10.0 l
Dispersion Fiber:	
Long staple fine	135.0 g
Short staple washed	90.0 g
AAA-glass	45.0 g
Surface Active Agent:	
AH-861	337.5 ml
Organic Binder:	
Resole (R-7)	157.5 ml

2. Resitron® Bonded Samples

H_2O:	10.0 l
Dispersion Fibers:	
Long staple fine	135.0 g
Short sample washed	90.0 g
AAA-glass	45.0 g
Surface Active Agent:	
AOS Slurry	10.0 ml
Organic Binder:	
Resitron®-Former	180.0 g
Hardner	90.0 g

G. Summary of Fabrication Process

Based on the development conducted thus far 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 three key steps is shown below:

(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.

(5) Foam formation by centrifugal pump

- 2 Gal. System - (43 GPM) - 15 mins. ~ 20 mins.
- 10 Gal. System - (89 GPM) - 15 mins. ~ 20 mins.

(10) Heat treat foam in oven

- Temperature ranges

  150°C - 30 mins ~ 2 hrs.

  250°C - 30 mins. 1 hr.

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 second contract period.

H. Characterization of Foam Specimens

The Fiberfrax® foams 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 Evaluation

The density of the foam specimen was determined by

dividing the total weight of the sample by the estimated volume. Generally speaking, the Resitron® bonded specimens appear to be less dense than the R-7 bonded counterparts. They also differ in color and foam structure. The following table shows the typical results from these two binders.

	Size	Fiberfrax® Blend (lbs/cu.ft.)	Fiberfrax®/AAA Blend/Glass (lbs/cu.ft.)	Remarks
Resole (R-7)	12" x 12"	0.814	0.821	Tan
	24" x 30"	1.050	1.100	Somewhat lumpy
Resitron® ∝-Resin	12" x 12"	0.853	0.847	Gray
	24" x 30"	0.771	0.774	Fine and homogeneous

2. Thermogravimetric Analysis

The purpose of this study was to investigate and compare the thermal behavior of Resole and Resitron® bonded foam. The samples studied were those of blended Fiberfrax®, long and short fibers, and 16-2/3% AAA glass fiber modified foam blend:

Resole - a. Foam skin of standard blend
b. Foam of standard blend
c. Foam skin of modified blend
d. Foam of modified blend

Resitron® - a. Foam skin of standard blend
b. Foam of standard blend
c. Foam skin of modified blend
d. Foam of modified blend

The TGA was carried out both in air and nitrogen atmos-

phere up to 800 °C and the thermograms are shown in Figures 3 through 6. The results indicate the following trends:

a. Standard resole formulation contains about 2% resin.

b. Modified resole formulation contains about 3% resin.

c. Significant reduction in resole contained in skin of standard formulation to about 6% from over 33% as was shown in Bimonthly Report No. 3.

d. Standard Resitron® formulation contains about 3% resin.

e. Modified Resitron® formulation contains about 4% resin.

3. Microscopie Examination

To ascertain the distribution of component fibers and the resin binder in the final foam structure the foam specimens were examined under the electron microscope. Figures 7 and 8 show the typical structure of resole and Resitron® bonded foams. In both cases, the distribution of fibers with different diameters appears to be homogeneous. The presence of resin in the fiber interface is obvious. It is significant to note that the resole tends to spread out as a fiber in the bonding area while the Resitron® resin tends to hold the fibers together via spot bondings. This observation explains the difference in resiliency in these two types of foams.

4. Wicking Test

The wicking test was performed according to the procedures described in Federal Specification CC-T-191b, Method 5830. The test procedure was originally developed by The Boeing Company. 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 different 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 $\pm$5°F. Note the degree of wicking every 24 hours.

Wicking After Oven Aging - Age the insulation material (approximately 8 x 14 inches), in a forced air circulating oven, at 160 $\pm$ 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.

Sample 443, a 24" x 30" unmodified Fiberfrax® foam sample, resole bonded, was tested in accordance with the above procedures. The average temperature for the oven aged sample was 162.5°F and 83.2°F for the leached sample. The wicking test apparatus used and degree of wicking are shown in Figures 9 and 10 respectively. Table 3 shows the average degree of wicking for the above samples.

The results show that all but 2 pieces of as received material did not wick above the allowable limit. Testing of the two pretreated samples shows that the average wicking exceeds the limit on most of the pieces tested. To improve this condition, investigation was made of a

post-treatment to make the foam water repellent. One method tried was a surface coating of Scotch-gard® and another method was a 2 step treatment with Scotchban® paper size FC805, a water soluble fluorochemical. Both products are manufactured by 3M Company.

The second method was chosen to post treat sample 559. First, three 8" x 14" 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.

Results of the "as received", post-treated, sample in the wicking test show that other than surface wetting no wicking occurred. In 2 of the pieces tested, isolated wicking occurs which may be due to improper post-treatment, since the complete cross section of the wicking area is not affected.

5. Flame Impingement Test

The flame impingement test for the Fiberfrax® foam specimens was carried out at NASA Lyndon B. Johnson Space Center, Structural Test Branch, Houston, Texas according to the standard procedures described in the "Test Plan for Flame Impingement of Aircraft Insulation Materials" prepared by Structures and Mechanics Division, Structural Test Branch, Manned Spacecraft Center, Houston, Texas (September 1, 1971). Two series of tests were conducted. The results are presented in Figures 11 and 12.

In Figure 11, the backface temperature of the Fiberfrax® foam insulator with a density of 0.8 lb/cu.ft. was compared with those of other competitive insulators. The result clearly showed that the Fiberfrax® foam was superior to the other systems with the same density level.

In Figure 12, test results for the full size (24" x 30") Fiberfrax® foam specimens were shown. The effectiveness of the foam insulators was demonstrated by the low backface temperature recorded throughout the entire test cycle, 10 minutes. It should also be mentioned here that the resole bonded specimen produced a very small amount of fume during the test and no fume was noticed in the Resitron® bonded foam. The difference in backface temperature between the Resitron" and resole bonded samples was mainly due to the difference in density.

6. Acoustical Test

The acoustical test of all the Fiberfrax® foams 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 entire contract period a number of specimens, both 12" x 12" and 24" x 30", were submitted to NASA for testing. The complete results are tabulated in Table 4.

It is obvious that the noise reduction of the foams developed thus far is not up to the satisfactory level yet. It is important to recognize our effort to improve the noise reduction by incorporating the fine diameter fibers such as Fiberfrax® fiber HF Grade and Johns-Manville's microfiber Grade AAA did indeed show a trend of improvement. Further efforts are needed in order to maximize the acoustical performance by optimizing the fiber composition and structure of the Fiberfrax® foam. This will be one of the major tasks to be accomplished in next year's program.

TABLE 1

Fiberfrax® Properties

Fiber Type	Composition Al_2O_3 %	Composition SiO_2 %	Fiber Diameter (Mean) μ	Fiber Length in.	Temperature Continuous Use °F	Temperature 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 μ	2300	3260	2.53
Milled	51.7	47.6	2-3	14 μ	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
Liquifoam[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 wicking per sample

Sample Condition	Avg. Temp. (°F)	Sample Taken from Length (in.)			Sample Taken from Width (in.)		
As received	78.8	0.250	0.375	0.375	0.250	0.250	0.250
	120.0	0.250	0.250	0.250	0.250	0.250	0.250
Oven Aged	71.9	0.375	0.340	0.290	0.250	0.290	0.260
	121.2	0.333	0.333	0.333	0.385	0.360	0.333
Leached	73.0	0.365	0.219	0.200	0.365	0.260	0.260
	121.4	0.354	0.302	0.260	0.333	0.292	0.250
Surface-* treated	73.1	0.000	0.000	0.000	0.000	0.000	0.000
	123.8	0.000	0.375	0.000	0.000	0.250	0.000

* These samples were post-treated with Scotchban FC-805, 2 step method. The isolated wicking of two of these post-treated samples appear to be due to improper application since it does not wick through complete area of pieces in question. Surface wetting occurs within allowable limits(1").

TABLE 4

Aircraft Acoustic Materials Test
Fiberfrax® Foam Insulation

Sample Number	Sample Designation and Composition	Density (lbs/cu.ft)	Size (inches)	Thickness (inches)	Noise Reduction, dB 1000 Hz	2000 Hz	4000 Hz
Control	LS/SSW	0.80	12 x 12	3.0	2.3	4.0	6.2
385	LS/SSW/FG	0.83	12 x 12	1.0	1.5	2.5	3.0
386	LS/SSW/KY	0.78	12 x 12	1.0	2.0	3.0	3.5
349	LS/SSW/KY	0.78	12 x 12	1.5	2.0	3.5	4.0
351	LS/SSW/KY	0.79	12 x 12	1.6	1.5	2.5	3.0
349/351	LS/SSW/KY	0.79	12 x 12	3.0	3.5	5.0	6.5
452	HF/LS	0.98	12 x 12	1.25	1.5	3.0	3.5
450	HF/LS	0.87	12 x 12	1.38	2.0	3.5	4.5
457	HF/LS	0.98	12 x 12	1.18	2.5	3.5	4.5
504	LS/SSW/AAA	0.77	12 x 12	1.75	0	4.0	7.0
506	LS/AAA	0.54	12 x 12	1.25	0	2.0	4.0
507	LS/SSW/AAA	0.60	12 x 12	1.63	0.5	2.5	5.0
* 509	LS/HF	0.83	12 x 12	1.50	0	2.0	5.5
* 510	LS/HF	0.93	12 x 12	1.38	0	4.5	7.0
* 512	LS/HF	0.81	12 x 12	1.38	1.0	2.5	5.0
* 520	LS/SSW/AAA	0.78	12 x 12	1.75	0	3.0	6.0
372	LS/SSW/KY	0.94	24 x 30	1.00	2.5	4.0	5.5
358	LS/SSW	1.62	24 x 30	0.75	3.5	4.5	6.0
368	LS/SSW	0.89	24 x 30	1.50	2.5	3.5	6.0
371	LS/SSW	1.13	24 x 30	1.18	2.5	4.0	5.5
368/371	LS/SSW	1.00	24 x 30	2.70	6.5	7.0	10.0
570	LS/SSW/AAA	1.21	24 x 30	1.125	4.0	7.0	8.0
571/571	LS/SSW/AAA	1.25	24 x 30	2.2	7.0	12.0	15.0
570/571/572	LS/SSW/AAA	1.25	24 x 30	3.3	10.0	18.0	23.0

TABLE 4 Cont'd.

Aircraft Acoustic Materials Test
Fiberfrax® Foam Insulation

Sample Number	Sample Designation and Composition	Density (lbs/cu.ft)	Size (inches)	Thickness (inches)	Noise Reduction, dB 1000 Hz	2000 Hz	4000 Hz
* 578	LS/SSW/AAA	0.75	24 x 30	1.75	4.0	6.0	7.0
* 578/583	LS/SSW/AAA	0.76	24 x 30	3.4	7.0	11.0	15.0
* 583/585	LS/SSW/AAA	0.77	24 x 30	3.25	7.0	11.0	15.0
Requirement		0.6		3.0	8.0	19.0	27.0
Fiberglass		0.6		3.0	11.0	20.0	29.0

Fiber Code

HF - HiFi
LS - Long Staple
SSW - Short Staple Washed
KY - KYNOL
FG - Glass Fiber (Owens/Corning Continuous Roving chopped into 1/8" Lengths)
AAA - Johns-Manville Micro Fiber Code AAA

Samples without "*" are bonded with R-7 Resin

Samples with "*" are bonded with Resitron® Alpha Resin

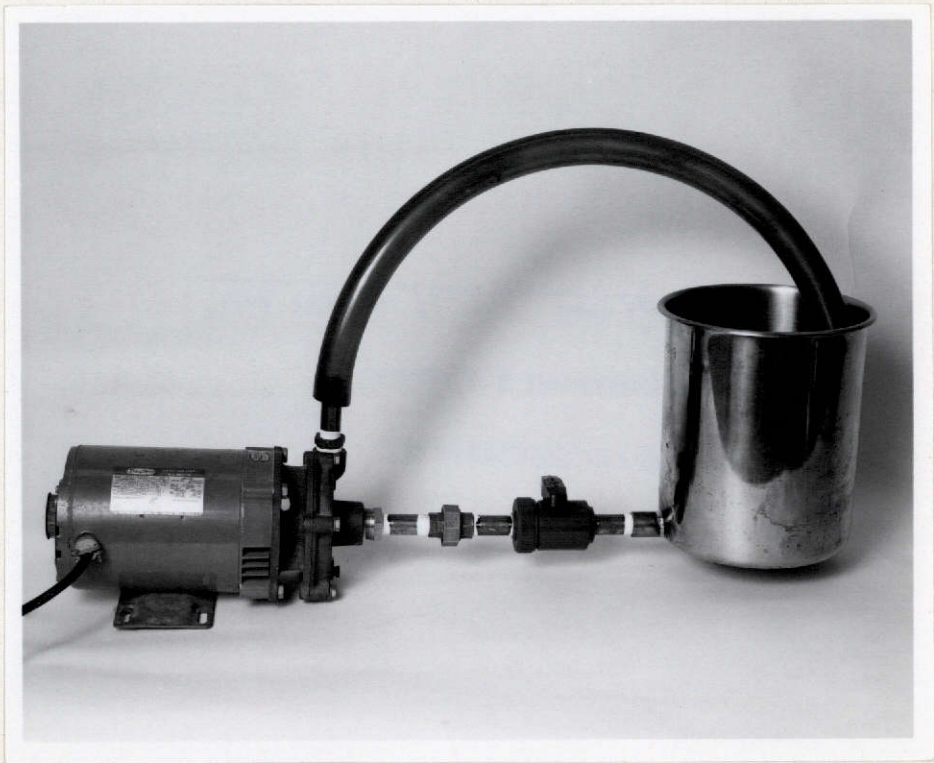


FIGURE 1

Two Gallon Foam Generator

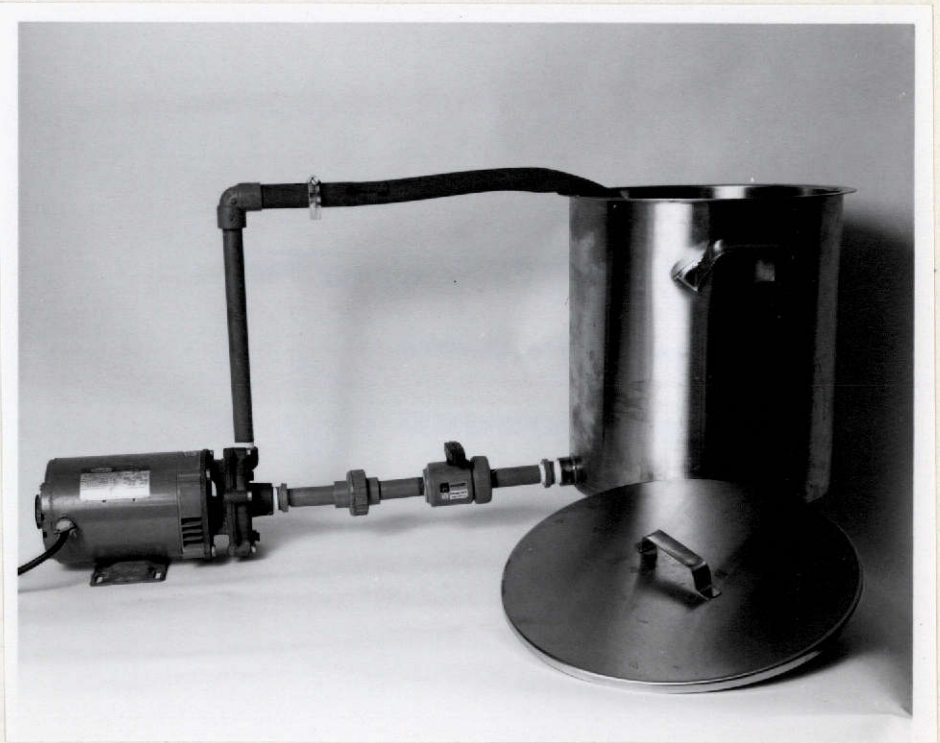


FIGURE 2

Ten Gallon Foam Generator

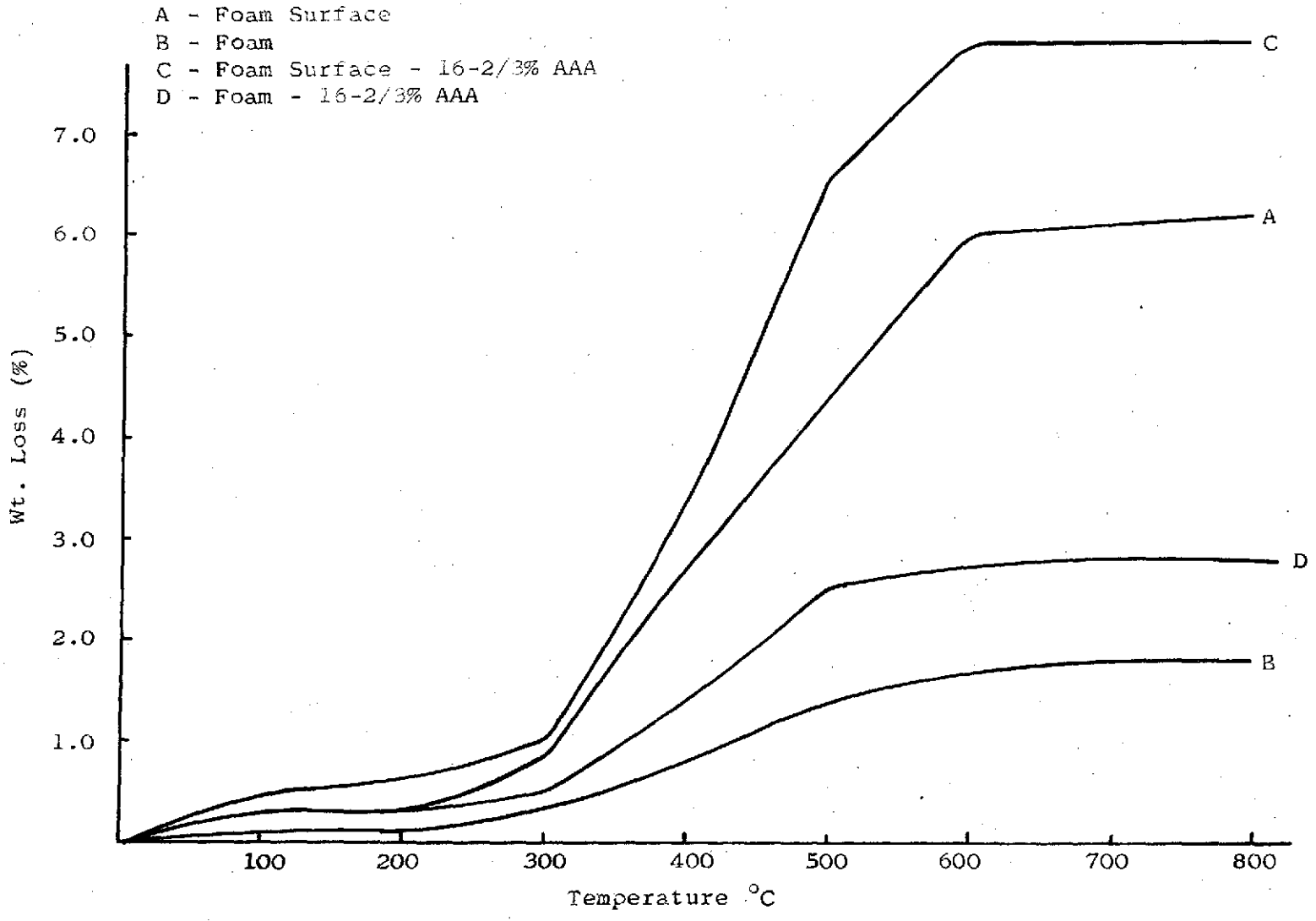


FIGURE 3

TGA of Foam Samples - Resole System in Air

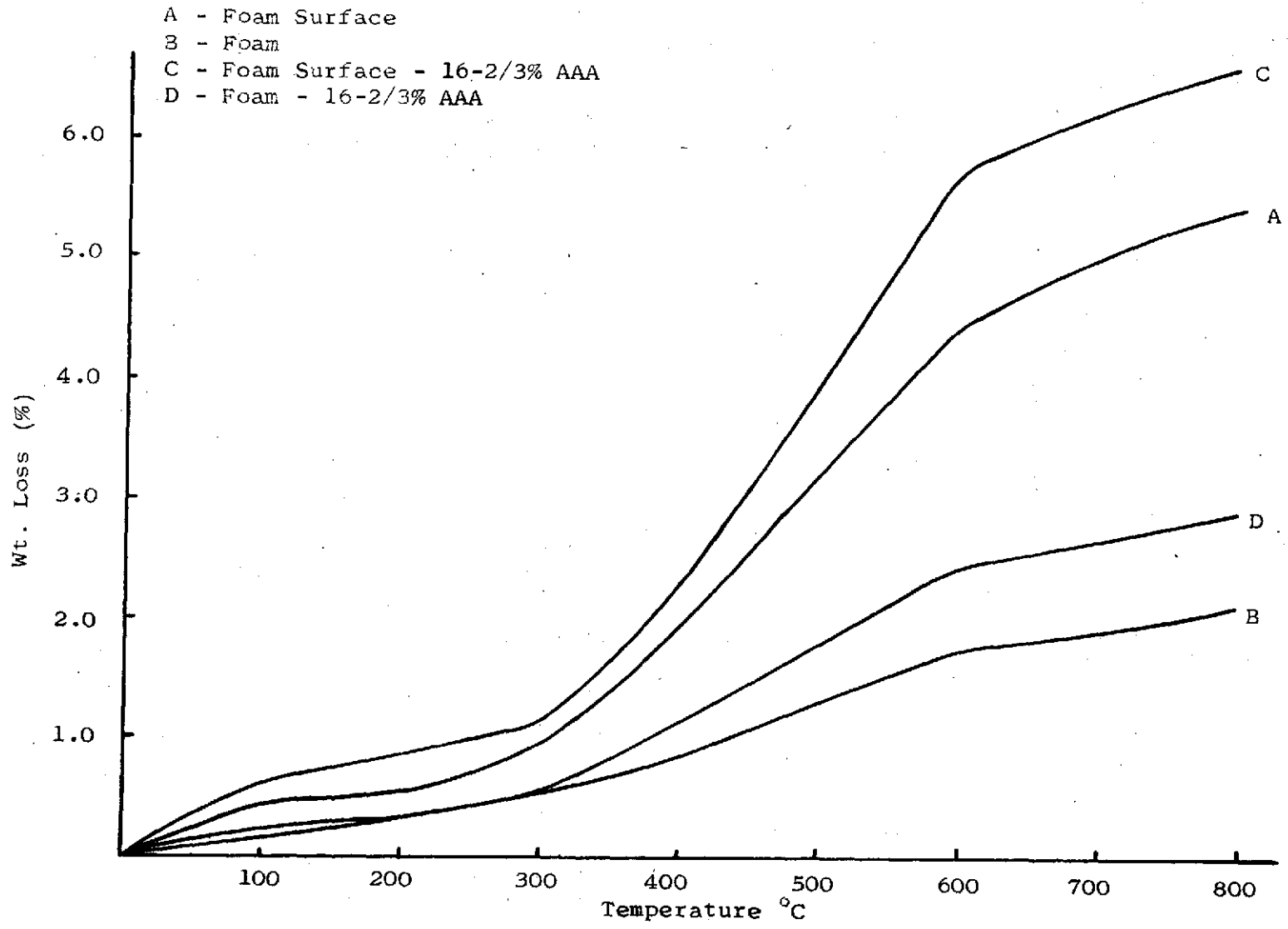


FIGURE 4

TGA of Foam Samples - Resole System
in Nitrogen Atmosphere

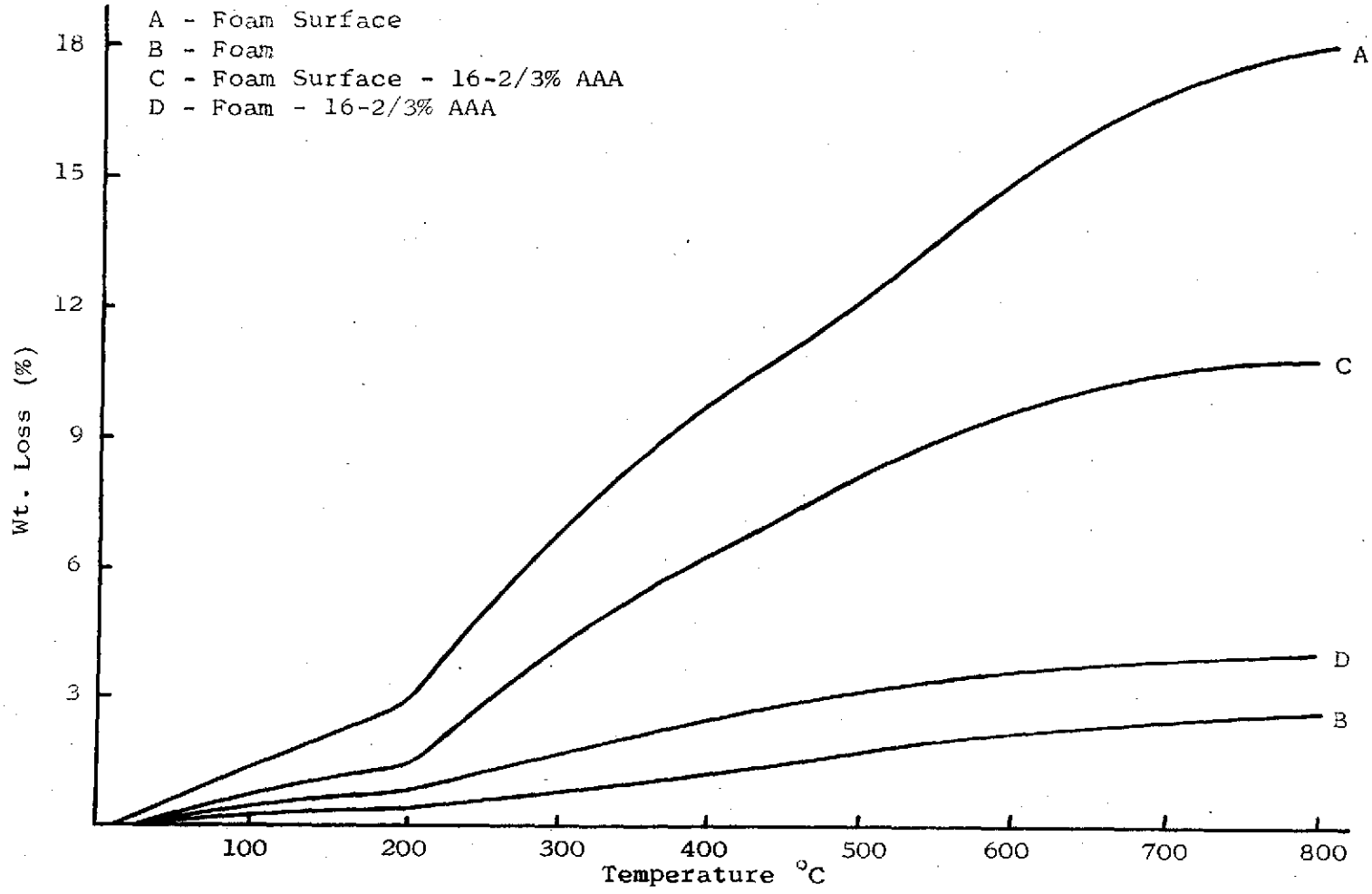


FIGURE 5

TGA of Foam Samples - Resitron® System in Air

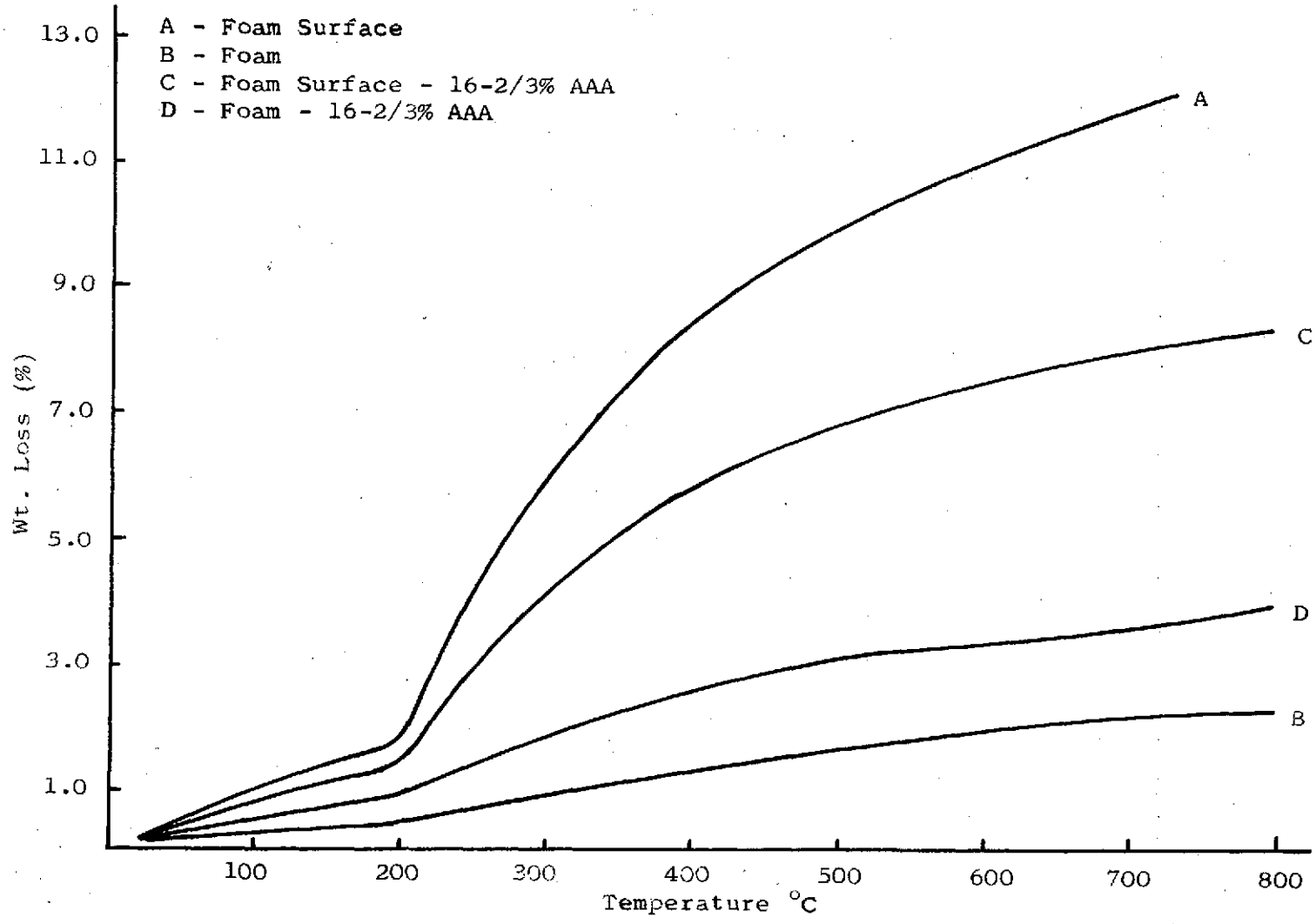


FIGURE 6

TGA of Foam Samples - Resitron® System
in Nitrogen Atmosphere

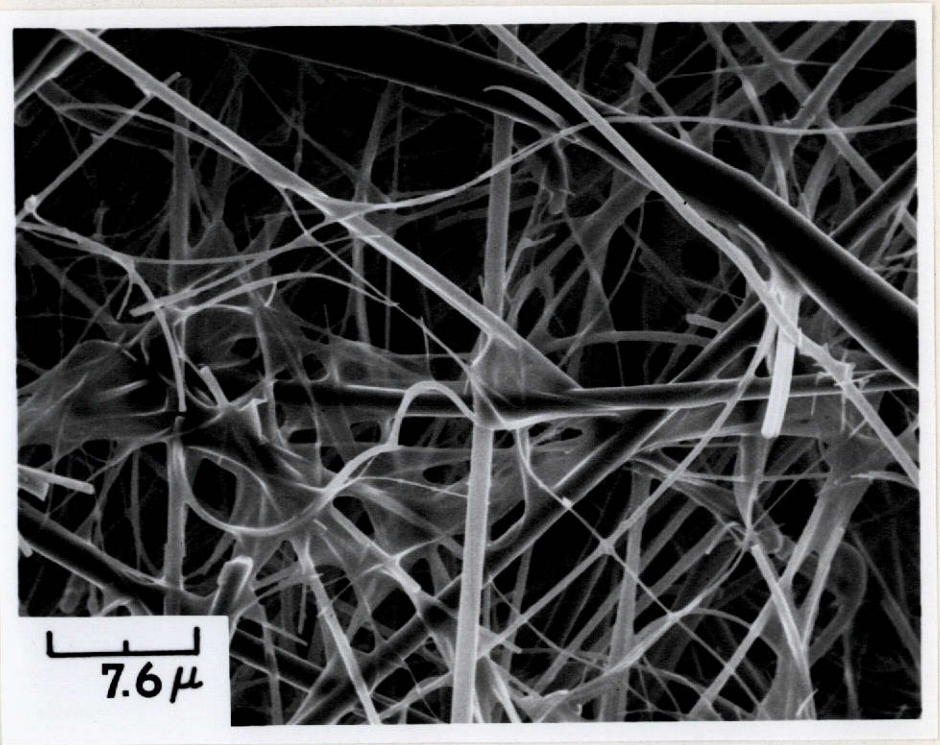


FIGURE 7

Electron Photomicrograph of Fiberfrax® Foam
Using Resole R-7 as Binder
(Sample No. C 586-8-576)

Sample Composition

45.0 g AAA Glass Fiber
135 g Long Staple Fiber) Fiberfrax®
90 g Short Staple Fiber)
382.5 ml Emulsifier AH 861
157.5 ml Resole R-7
10 l Water

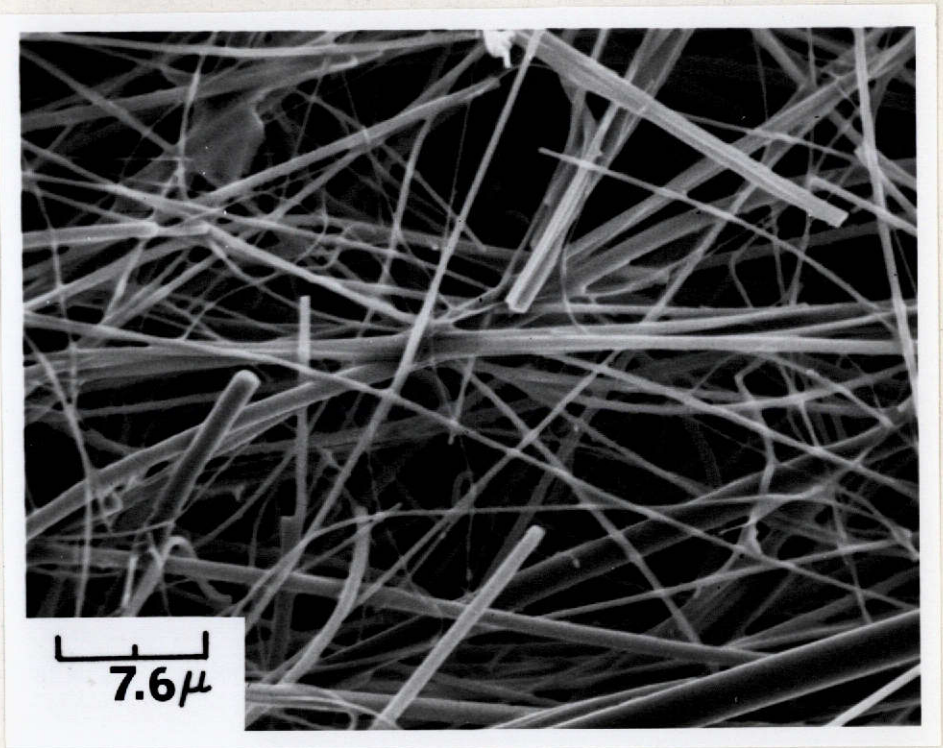


FIGURE 8

Electon Photomicrograph of Fiberfrax® Foam
Using Resitron® Resin as Binder
(Sample No. C517-87-519)

Sample Composition

9.0 g	AAA Glass Fiber	
27 g	Long Staple Fiber)	Fiberfrax®
18 g	Short Staple Fiber)	
2.7 ml	Sulframin AOS Slurry	
45.0 g	Resitron® Former	
22.5 g	Resitron® Hardener	
2 l	Water	

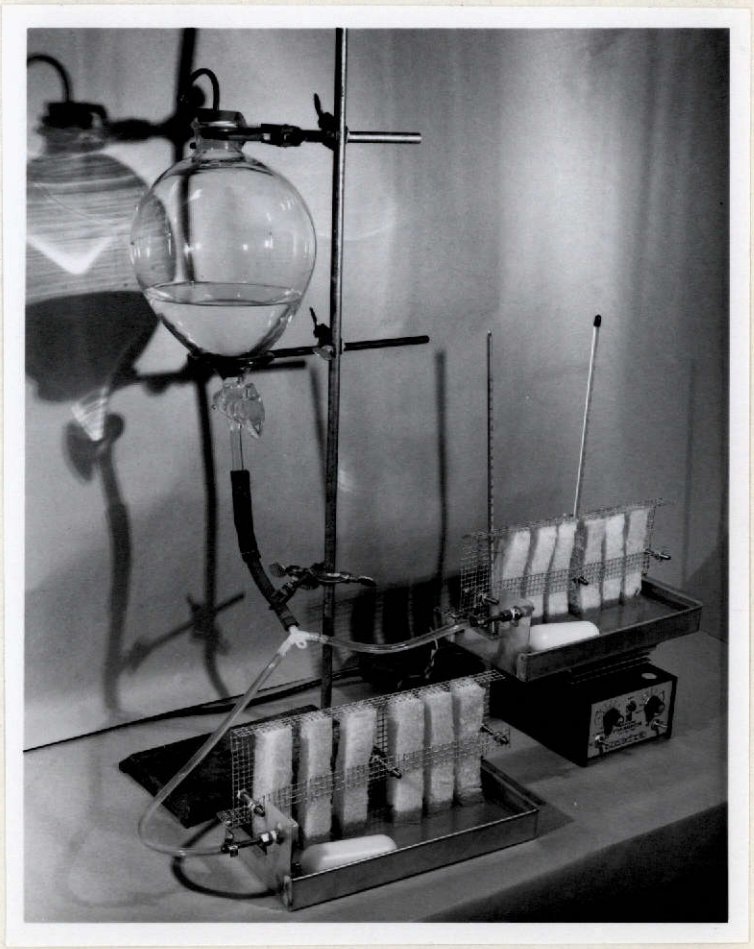


FIGURE 9

Laboratory Set-up for Wicking Test

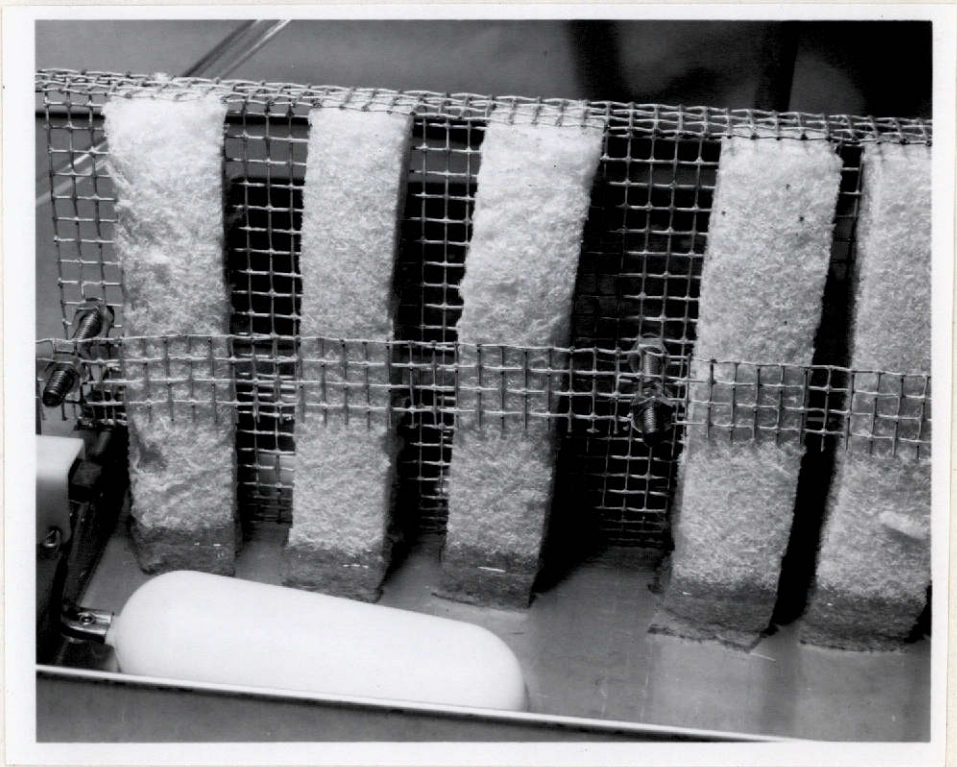


FIGURE 10

Degree of Wicking of Fiberfrax® Foams

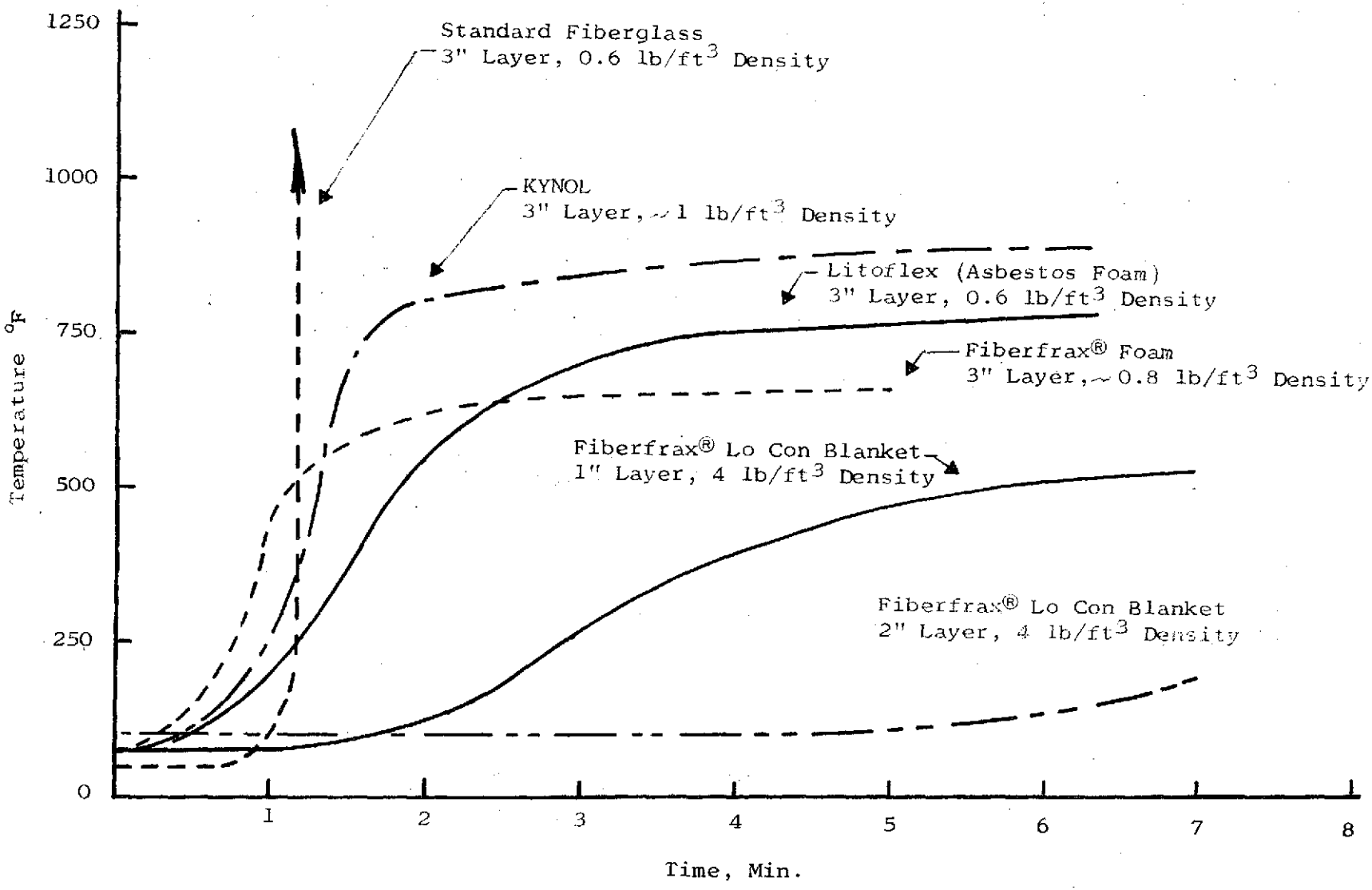


FIGURE 11 - Flame Impingement Tests for Various Insulators

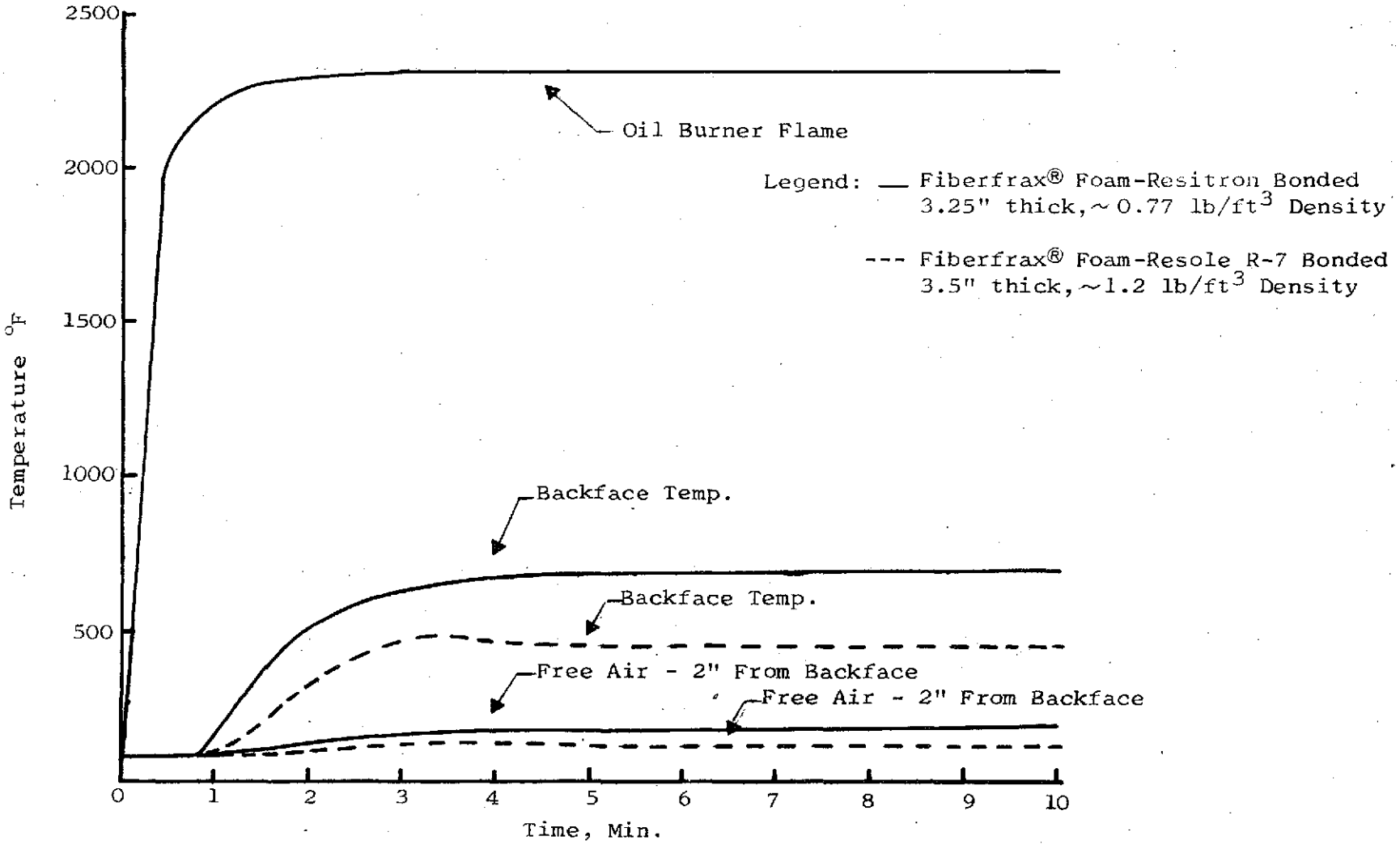


FIGURE 12 - Flame Impingement Test for Full Size (24" x 30") Fiberfrax® Foams

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