NASA CR-134805

DEVELOPMENT PROGRAM TO PRODUCE MULLITE FIBER INSULATION

by

W. G. Long

THE BABCOCK & WILCOX COMPANY

(NASA-CR-134805) DEVELOPMENT PROGRAM TO PRODUCE MULLITE FIBER INSULATION (Babcock and Wilcox Co., Augusta, Ga.)

N75-22384

Unclas

G3/24 19433

Prepared for

NATIONAL AERONAUTICS & SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-16782
John P. Merutka, Project Manager

April 1975

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U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

1. Report No. CR-134805	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle DEVELOPMENT PROGRAM TO PRODUCE MULLITE FIBER INSULATION	5. Report Date April 4, 1975
	6. Performing Organization Code
7. Author(s) W. G. LONG	8. Performing Organization Report No.
	10. Work Unit No.
9. Performing Organization Name and Address THE BABCOCK & WILCOX COMPANY REFRACTORIES DIVISION OLD SAVANNAH ROAD AUGUSTA, GEORGIA THE BABCOCK & WILCOX COMPANY RESEARCH & DEVELOPMENT DIVISION P. O. BOX 1260 LYNCHBURG, VIRGINIA	11. Contract or Grant No. NAS 3-16782
	13. Type of Report and Period Covered CONTRACTOR REPORT
12. Sponsoring Agency Name and Address NATIONAL AERONAUTICS AND SPACE ADMINISTRATION WASHINGTON, D.C. 02546	14. Sponsoring Agency Code

15. Supplementary Notes

PROJECT MANAGER, JOHN P. MERUTKA, NASA LEWIS RESEARCH CENTER, CLEVELAND, OHIO

16. Abstract

Two processing methods were utilized to form a mullite fiber-Kaowool fiber felt. The formation of a blended felt using the Rotoformer wet laying method was successful. Felt products were evaluated for tensile strength, thermal stability, thermal conductivity and structural integrity at 1259 C and 1371 C. Techniques common to the textile industry were utilized in an attempt to form a yarn from staple and multifilament mullite fiber. All of the textile processing methods failed due to damage induced in the fiber through mechanical handling. The refractoriness of pure Kaowool ceramic fiber is improved with additions of 30 percent or greater mullite fiber.

PRICES SUBJECT TO CHANGE

17. Key Words (Suggested by Author(s)) CERAMIC FIBER THERMAL PROPERTIES MULLITE/KAOWOOL FIBER BLENDED FELTS MULLITE FIBER	18. Distribution Statement UNCLASSIFIED - UNLIMITED
19. Security Classif. (of this report) UNCLASSIFIED	20. Security Classif. (of this page) UNCLASSIFIED

* For sale by the National Technical Information Service, Springfield, Virginia 22151

NASA-C-168 (Rev. 6-71)

FOREWORD

This report covers work performed under contract NAS 3-16782 for the period 1 January 1973 through 30 November 1974. The work was administered by the NASA-Lewis Research Center with Mr. John P. Merutka as Project Manager.

Research efforts were performed at the Lynchburg Research Center of Babcock & Wilcox under the direction of Mr. W.G. Long. Dr. A.V. Illyn, Technical Director of the Refractories Division, served as project director for this contract.

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1. SUMMARY

Mullite fiber and Kaowool fiber, both in staple form, were processed by a wet laying method to produce a series of blended felts. The felts were produced in the following mullite to Kaowool fiber ratios by weight: 90/10; 75/25; 45/55; 30/70; 10/90; 0/100.

The refractoriness, or dimensional stability at elevated temperature, was improved for all levels of mullite fiber above 10 percent as compared to a 100 percent Kaowool insulating blanket product. Table I summarizes the thermal shrinkage produced in felted specimens after 25 thermal cycles to 1259C and to 1371C.

Table I. Thermal Shrinkage of Felted Products

Felt Blend	Percent Linear Shrinkage	
	After 1259C	After 1371C
1. 90 mullite/10 Kaowool	0	-4
2. 75 mullite/25 Kaowool	1	2
3. 45 mullite/55 Kaowool	1	2
4. 30 mullite/70 Kaowool	2	3.2
5. 10 mullite/90 Kaowool	5	13.5
6. 100 Kaowool	5	14

It can be seen from the results in Table I that the 30 weight percent and 45 weight percent mullite fiber felts have excellent dimensional stability after exposure to 1371C. A product combining mullite and Kaowool ceramic fiber in these proportions would increase the useful life of a 100 percent Kaowool product by at least 110°C.

The thermal conductivity of three blended felt products tested was not significantly different from a standard 6 pcf Kaowool ceramic blanket product. A slight increase in the conductivity was experienced relative to Kaowool fiber blanket, but this can be attributed to the lower density of the felted products.

The handleability of all of the felts as produced was good. After the thermal exposure the handleability and tensile strength of all of the felts was very poor relative to Kaowool fiber blanket.

The Rotoformer method of forming felted products has the flexibility and capacity for high volume rates of production of a blended fiber product on a continuous basis.

The feasibility of forming a felted product from mullite and Kaowool fiber by conventional textile processing methods was evaluated. In all cases, the mechanical damage to the mullite fiber was excessive. These results are summarized in Appendices I and II.

2. INTRODUCTION

The original objective of this program was to develop a mullite yarn with properties suitable for use on space shuttle applications. Possible uses for such a product include a reinforcement for insulating tiles, a back-up thermal insulation behind a leading edge and packing for wheel wells. Fabric Research Laboratories, Dedham, Massachusetts provided textile equipment and fabrication experience on a subcontract basis for this program.

Working with staple fiber and multifilament mullite fiber tows, several textile processes were utilized and all attempts were unsuccessful. The stiffness and low elongation to failure of mullite fiber made the incorporation into a yarn appear to be an impossible task. The carding of the mullite fiber reduced the individual fiber lengths to less than 1/2". A modified carding system, the Shirley Method, which employs a slower carding operation, was evaluated with and without an organic fiber carrier, and this method also destroyed the fiber. Based upon the results using the Shirley Method and with scavenger yarns, the feasibility of forming a mullite yarn was judged to be poor.

A blend of mullite fiber with Babcock & Wilcox Kaowool fiber, using less than 5% polyester fiber as a carrier, was evaluated and showed considerable promise. A blend of 91 w/o mullite - 4.5 w/o Kaowool with 4.5% polyester was thermal cycled and showed less than 2% shrinkage at 1259C.

Based upon this evaluation, a decision was made to modify the contract, showing a revised Task III which required the evaluation of a series of mullite fiber - Kaowool fiber felted blends before and after thermal exposure to 1259 C and 1371 C.

3. EXPERIMENTAL PROCEDURE

3.1 Yarn Properties

The target properties for the yarn produced from mullite fiber are as follows:

(1) Yarn as-processed must be capable of withstanding a tensile force of $\geq$ 9 kgm without fracture.

(2) After exposure to 1260°C in one atmosphere of dry air for 25 hours, the yarn must be capable of withstanding a tensile force of $\geq$ 4.5 kgm.

(3) Yarn as-processed must be capable of being bent 180° on a maximum radius of 9.5 mm without failure.

(4) The linear density of the yarn must be $\leq$ 4.0 mgm/cm.

(5) The length of the yarn must not be less than 65 cm.

Testing of any yarn produced will be conducted to determine whether it meets the above requirements and whether the results are reproducible.

3.2 Materials

The mullite fiber utilized in this project was fiberized from solution into two distinct fiber types. The B&W mullite fiber, which has been developed and reported earlier,(1,2) has an average fiber diameter of 5 microns and is described as a staple fiber. The other mullite fiber is a continuous extruded form and is collected in a tow and is identified as multifilament fiber. The chemistry of the two fibers is identical and is tested as the standard B&W mullite composition as follows:

Oxide Constituent	Wt. %
Al_2O_3	77
SiO_2	17
B_2O_3	4.5
P_2O_5	1.5

Kaowool ceramic fiber, a B&W alumina-silica product, was combined with mullite fiber in the later stages of this program. Kaowool is a high temperature commercial fiber insulation with a chemistry as given below. The fiber diameter distribution in Kaowool is similar to that in mullite fiber.

Kaowool Ceramic Fiber Compsotion

	wt %
Al_2O_3	45
SiO_2	52
FeO	1.3
TiO_2	1.7

The five textile techniques used (listed and described in Appendix I) to process mullite fiber into a yarn were all unsuccessful. The Rotoformer wet laying method was successful in combining Kaowool and mullite fibers into a wet laid felt.

3.3 Materials Processing

Wet-Laid Felt - The paper and pulp industry typically manufactures products from a water slurry on equipment which lays a metered amount of slurry on a moving belt or drain which is vacuum equipped to evacuate water. Typical examples of these types of equipment are the Fourdrinier and the Rotoformer. The latter is shown schematically in Figure 1, and was used to produce blended Kaowool-mullite felted products in the following weight percents: 10 Kaowool-90 mullite; 25 Kaowool-75 mullite; 55 Kaowool-45 mullite; 70 Kaowool-30 mullite; and 90 Kaowool-10 mullite. These blends were felted and compared to a commercial B&W ceramic fiber blanket which is 100 percent Kaowool fiber.

The Rotoformer used in this felting operation is located at the Sandy Hill Corporation, Hudson Falls, New York. The fiber was first mixed in large tanks to a concentration of approximately 1 part fiber to 99 parts water, by weight. The fiber-water slurry was fed into the Rotoformer and by manipulating the flow, and spacing settings, a 1-1-1/2" thick felted product was continuously formed. The felts were then dried in an air oven at 150 F and shipped to the Lynchburg Research Center of B&W for evaluation.

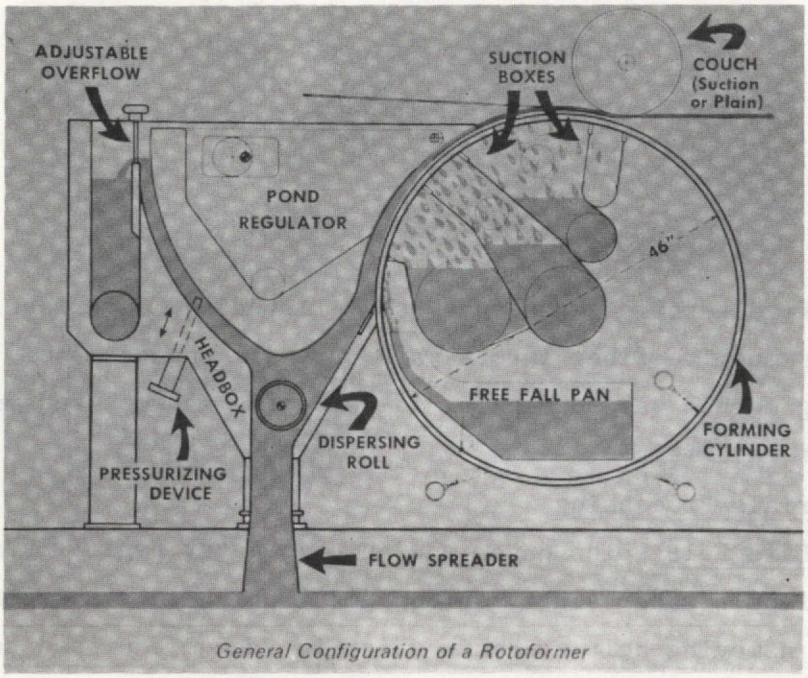

FIGURE 1. Schematic Showing Material Feed and Flow, and Product Formation on the Forming Cylinder.

The evaluation consisted of measuring the density, dimensions, resiliency, and tensile strength before and after exposure to 25 thermal cycles between room temperature and both 1259 C and 1371 C. Further evaluation included thermal conductivity measurements on two compositions as fabricated and following 1371 C and an observation of the handleability and structural integrity before and after thermal exposure.

4. RESULTS AND DISCUSSION

The application of textile processing to mullite fiber to produce a yarn product was unsuccessful. Fibers were processed into several forms including paper felt, slivers, webbing, and thick batting. The individual unsuccessful processes examined to form mullite fiber into yarn and the results produced are summarized in Appendix II.

Task III of the contract was revised to evaluate a series of mullite/ Kaowool fiber felt blends which were successfully made. The following properties were examined and are reported.

4.1 Tensile Strength

The tearing strength of a felted or blanket product is normally determined on a wide piece of material to obtain some measure of the integrity of the product for handling and installation. In these tests, an 8-inch wide by 6-inch long specimen was cut from each blend composition and tested in a fixture as shown in Figure 2. A 6-inch width of product is loaded in tensile and the fracture or tearing strength is recorded.

The results of the tensile tests on the mullite-Kaowool felted products are given in Table II. The high mullite compositions generally failed at a load of 1 pound or less. The 55 and 70 percent Kaowool felt supported a 2 pound load and the 90 Kaowool - 10 mullite failed under a 3 pound load. Following exposure of 25 thermal cycles to 1259 C and 1371 C, the felts all failed in placing the test samples in the test fixture. The felts seemed to be brittle after the high temperature exposure. A method of support and contaiment would be required to use these felted products in normal refractory applications. A more practicable approach would be to introduce an inorganic binder system to produce a semi-rigid product with increased structural integrity.

4.2 Thermal Stability

The density values of the felted products fabricated on the Rotoformer are given in Table III. The as-fabricated density ranged from 2.9 pounds per cubic foot (pcf) for the 10 Kaowool - 90 mullite to 5.5 pcf for the 90 Kaowool - 10 mullite products. The lower densities are related to the relative stiffness of the mullite fiber, making consolidation into a dense product very difficult. Previous experience at B&W with vacuum forming a pure mullite fiber composition and a colloidal

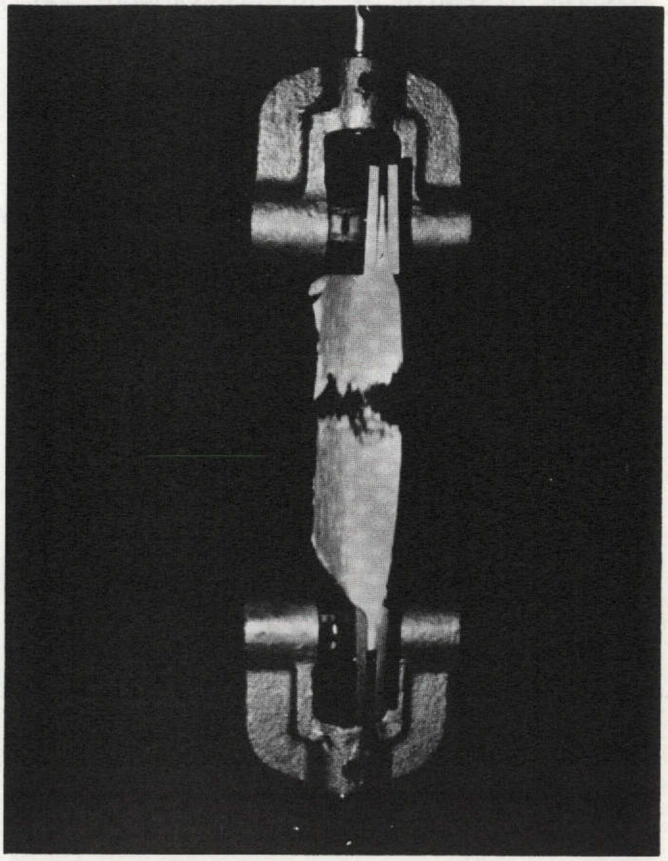

FIGURE 2. Fixture Used to Measure Tensile Strength of a Felted or Blanket Insulating Product. (A Six-Inch Width of Material is Being Tested.)

TABLE II. TENSILE STRENGTH OF MULLITE-KAOWOOL FELTED PRODUCTS

		As-Fabricated lbs.*	After 1259 C Exposure, lbs.	After 1371 C Exposure, lbs.
1.	10 Kaowool 90 mullite	would not support a load in any of the tests		
2.	25 Kaowool 75 mullite	1	0	0
3.	55 Kaowool 45 mullite	2	0	0
4.	70 Kaowool 30 mullite	2	0	0
5.	90 Kaowool 30 mullite	3	0	0
6.	100 Kaowool (6 pcf commercial blanket)	20	24	14

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*Strength defined on a 6 inch width of felt, with the breaking load recorded in pounds.

TABLE III. CHANGE IN DENSITY OF MULLITE-KAOWOOL FELT PRODUCTS WITH ELEVATED TEMPERATURE EXPOSURE

	As-Fabricated, pcf	After 1259 C pcf	After 1371 C pcf
1. 90 Mullite 10 Kaowool	2.9	2.8	2.3
2. 75 Mullite 25 Kaowool	4.9	5.0	5.2
3. 45 Mullite 55 Kaowool	4.7	4.8	5.1
4. 30 Mullite 70 Kaowool	5.2	5.3	5.4
5. 10 Mullite 90 Kaowool	5.5	6.3	8.2

silica binder indicated that a 9 pcf product could be formed. Using a Kaowool ceramic fiber in the same process would result in a 12-18 pcf product.

The 10 Kaowool - 90 mullite felt decreased in density following elevated temperature exposure. This is undoubtedly caused by the inclination of the mullite fiber to return to a stress-free condition. The fiber uniformity of the low density (2.9 pcf) felt is not good, so conclusions based upon these results are tentative. However, there is a pronounced tendency for the low density (high mullite) felts to expand at temperatures of 1259 C and 1371 C.

The 55 Kaowool - 45 mullite and the 70 Kaowool - 30 mullite fiber felted products increased in density by 8.5 and 3.8 percent, respectively, after the 1371 C exposure. The 1259 C thermal cycling produced approximately a 2 percent increase in density. These values are low and the blends containing 30 percent and greater mullite fiber are all considered thermally stable for repeated exposures to 1371 C.

The 90 Kaowool - 10 mullite showed a marked increase in density from 5.5 pcf to 6.3 pcf after 1259 C and to 8.2 pcf following the 1371 C thermal cycling. This blend has insufficient mullite to provide the additional refractory service above 1259 C, the nominal upper use limit temperature for Kaowool ceramic fiber insulation.

The linear shrinkage measured over a 5 inch gage length of felt material is given in Table IV. Following the 1259 C exposure less than 2% linear shrinkage was noted on all the felted products of 30 percent or greater mullite fiber. The 10 percent mullite fiber addition to Kaowool provided no benefit in reducing thermal shrinkage either at 1259 C or at 1371 C. The shrinkage noted for both of these temperatures was consistent with that produced in a 6 pcf Kaowool insulating blanket product, as shown in Table IV.

After 25 thermal cycles to 1371 C, the shrinkage produced was 3.2 percent for the 30 mullite - 70 Kaowool felt. All of the higher mullite compositions showed less shrinkages. Based upon these results, the felted products with 25 or greater percent mullite would be considered suitable for repeated exposure at 1371 C. The 90 mullite - 10 Kaowool is a special case where the density is too low to be seriously considered as thermal insulation, as noted above.

TABLE IV. THERMAL SHRINKAGE OF MULLITE-KAOWOOL FELTED PRODUCTS

		Original Dimension, inches	After 1259 C Exposure, inches	After 1371 C Exposure, inches
1.	10 Kaowool 90 Mullite	5	5 (0% shrinkage)	5.2 (4% growth)
2.	25 Kaowool 75 Mullite	5	4.95 (1% shrinkage)	4.9 (2% shrinkage)
3.	55 Kaowool 45 Mullite	5	4.95 (1% shrinkage)	4.9 (2% shrinkage)
4.	70 Kaowool 30 Mullite	5	4.90 (2% shrinkage)	4.54 (3.2% shrinkage)
5.	90 Kaowool 10 Mullite	5	4.75 (5% shrinkage)	4.32 (13.5% shrinkage)
6.	100 Kaowool (6 pcf blanket)	5	4.75 (5% shrinkage)	4.30 (14% shrinkage)

4.3 Thermal Conductivity

The thermal conductivity was determined according to ASTM procedure C201, modified to include silicon carbide divider plates as shown in Figure 3. The heat flow is measured in the calorimetry circuit in units of BTU/hr for an area of insulation expressed in square feet, insulation thickness in inches, and a thermal gradient in degrees Fahrenheit. The units, as expressed in Table V and Figures 3 and 4, are $\text{BTU-hr}^{-1}\text{-ft}^{-2}\text{-in.}^{-1}\text{-}^{o}\text{F}^{-1}$. The values shown in Table V and presented in Figure 4 show very little difference between the 30 mullite - 70 Kaowool and the 45 mullite - 55 Kaowool fiber blended products. Exposure to temperatures to 1371 C produce no notable change in the conductivity values.

The conductivity of the two blended products tested was higher than the values produced on a 6 pcf Kaowool blanket product, as shown in Figure 4. This may be related to the lack of uniformity caused by the rigidity of the individual mullite fibers. The density of the fiber insulation is an overriding consideration, with insultating efficiency increasing directly with density. The 6 pcf nominal density Kaowool blanket had an actual density of 6.2 pcf, which was significantly higher than the felted products tested, which ranged from 2.9 to 5.5 pcf.

4.4 Handleability and Structural Integrity

Thermal insulation must have sufficient strength to allow handling during installation and also to remain in place under stresses imposed in service. Generally, a rating of handleability or structural integrity is a subjective evaluation. Beyond the tensile strengths as presented in Table II, the handleability values are not normally quantified. Table VI lists the evaluations made on the felted products made on the Rotoformer. Using Kaowool commercial 6 pcf insulating blanket as a standard, none of the felted blends produced approached Kaowool blanket for handleability. The differences in handleability were especially noticeable following elevated temperature exposure. Kaowool insulating blanket retains a significant degree of its original handleability, flexibility, and integrity. Handleability increases in the felted products in this project as the percentage of Kaowool is increased. It would appear that all of the products above 55 weight percent Kaowool can be handled for installation purposes.

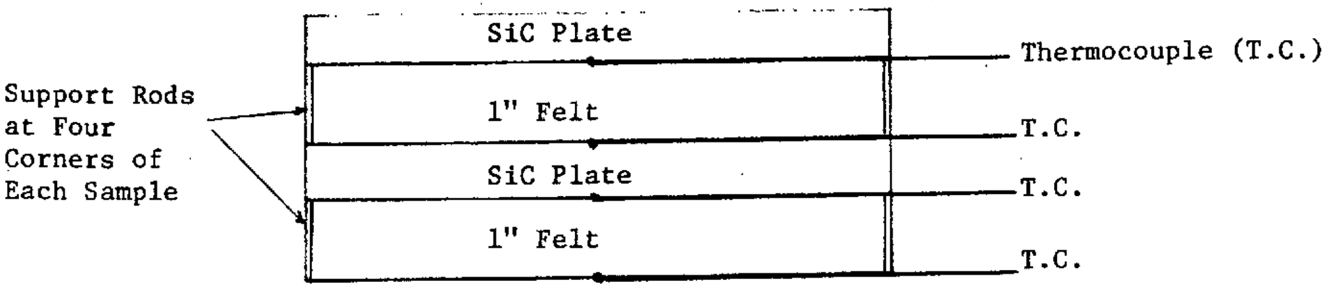

FIGURE 3. Schematic of Sample Arrangement in Thermal Conductivity Apparatus.

$$K = Q.A. \left(\frac{\Delta T}{\Delta X} \right)$$

where Q.A = BTu/hr/ft^2 which is the heat flow measured in the calorimetry circuit per square foot of sample area.

$\frac{\Delta T}{\Delta X}$ = thermal gradient in ^{o}F (ΔT) across a thickness of 1 inch (ΔX) as measured by the thermocouples in contact with the specimen.

TABLE V. THERMAL CONDUCTIVITY VALUES FOR MULLITE-KAOWOOL FELT PRODUCTS

Mean Temp., °F	K values in BTU-hrs^{-1}ft^{-2}-in^{-1}-^{o}F^{-1}		
	30 Mullite 70 Kaowool	30 Mullite 70 Kaowool	45 Mullite 55 Kaowool
		After 1371C Exposure	
800	.75	.78	.77
900	.85	.88	.87
1000	.96	1.00	1.00
1100	1.08	1.13	1.23
1200	1.21	1.26	1.27
1300	1.35	1.41	1.42
1400	1.50	1.57	1.58
1500	1.65	1.73	1.74
1600	1.81	1.89	1.91
1700	1.97	2.06	2.08
1800	2.14	2.24	2.26
1900	2.31	2.41	2.43
2000	2.48	2.58	2.61
2100	2.64	2.76	2.79
2200	2.82	2.94	2.97

Density of Test Specimens: 30 Mullite-70 Kaowool As-Fabricated = 5.6 pcf
30 Mullite-70 Kaowool After 2500 F = 6.2 pcf
45 Mullite-55 Kaowool As-Fabricated = 5.2 pcf

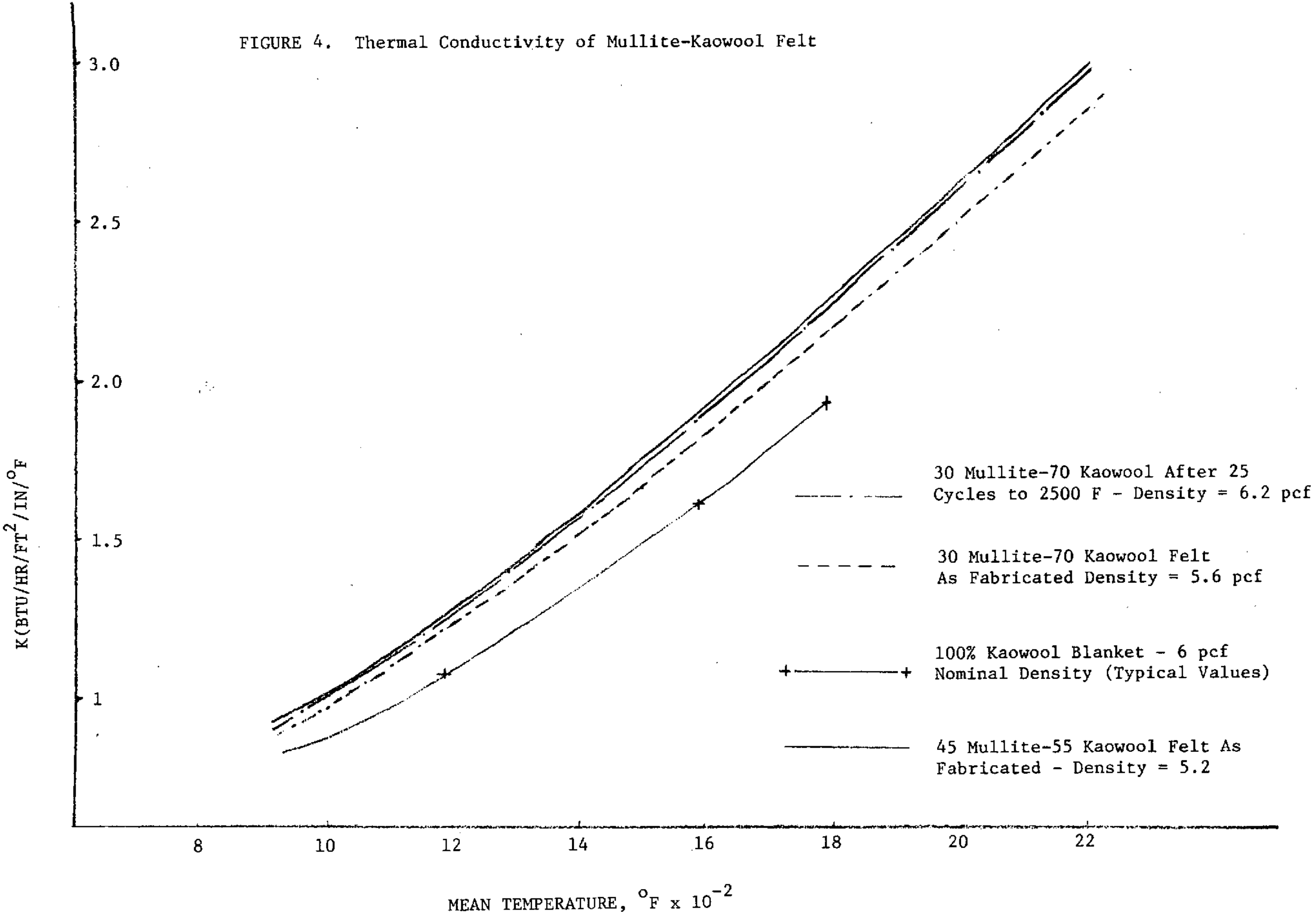

FIGURE 4. Thermal Conductivity of Mullite-Kaowool Felt

TABLE VI. HANDLEABILITY AND STRUCTURAL INTEGRITY OF MULLITE-KAOWOOL FELT PRODUCTS

		As-Manufactured	After 1259 C and 1371 C Thermal Exposure
1.	10 Kaowool 90 Mullite	low density (2.9 pcf) poor handleability pulls apart on handling	poor handleability decrease in density following exposure
2.	25 Kaowool 75 Mullite	fair handleability can be handled without causing damage	poor handleability severe damage in flexing
3.	55 Kaowool 45 Mullite	Moderate handleability can be handled without causing damage	poor handleability severe damage in flexing
4.	70 Kaowool 30 Mullite	moderate handleability flexing reduces integrity	handling causes severe damage on flexing
5.	90 Kaowool 10 Mullite	good handleability	handling causes severe damage on flexing
6.	100 Kaowool commercial 6 pcf blanket	excellent handleability strength, and flexibility	good handleability flexibility reduced, especially after 1371 C exposure

The resiliency of the mullite-Kaowool fiber felts was measured by compressing layers of the felted material to a dimension of 0.7 inches between parallel plates and then exposing the compressed sample to 815 C for 18 hours. The test was conducted in a muffle furnace with flowing argon as a cover gas. The dimensions of the starting specimens and the amount of springback, or resiliency, are shown in Table VII. The springback is determined by measuring the thickness of the felt after removing the top plate.

The variability in thickness of a single layer of the felt is shown in the free standing dimensions of the as-manufactured product. Very little springback is noted in these tests, due to the low density of the felt. In the tests of felt which had been exposed to 1259 C and 1371 C thicker specimens were formed by stacking 2-4 layers of felt. When compressed to 0.7 inches, the density of these specimens was 2-3 times greater than the specimens in the as-manufactured tests.

The results of the springback measurements shows the high mullite felts to be the most resilient. The 90 mullite - 10 Kaowool had a springback of 0.50 inches and 0.80 inches, respectively, for the felts exposed to 1259 C and 1371 C. The mullite fiber contributes significantly to springback, or resiliency, as demonstrated by comparing the values of the felts following elevated temperature exposure. In those applications where springback from a compressed condition is important, the addition of 45 or greater percent of mullite fiber will assure that resiliency is produced.

4.5 Potential for Scale-Up

The felts which were produced on the Rotoformer can be routinely processed at 40 square feet per minute. No further scale-up would be required for a profitable commercial application. All of the felted products in this program were produced on the Rotoformer at that rate.

The manufacturing characteristics of the products were similar, with batch mixing and feeding consistent for all products. The handling characteristics of the finished products would tend to favor the higher Kaowool fiber compositions. Maintaining a consistent thickness and density would also be more feasible with the 55 and 70 weight percent Kaowool.

TABLE VII. RESILIENCY OF MULLITE-KAOWOOL FELTED PRODUCTS

		As-Manufactured		After 25 Cycles to 1259 C		After 25 Cycles to 1371 C	
1.	10 Kaowool	free standing	1.5"	free standing	3.8"	free standing	4.8"
	90 Mullite	compressed to	0.7"	compressed to	.70"	compressed to	0.7"
		returned to	.79"	returned to	1.20"	returned to	1.5"
		springback	.09"	springback	.50"	springback	.80"
2.	25 Kaowool	free standing	1.2"	free standing	3.5"	free standing	2.5"
	75 Mullite	compressed to	0.70"	compressed to	.70"	compressed to	0.7"
		returned to	.76"	returned to	.95"	returned to	1.2"
		springback	.06"	springback	.25"	springback	.50"
3.	55 Kaowool	free standing	1.1"	free standing	2.8"	free standing	2.0"
	45 Mullite	compressed to	.70"	compressed to	.70"	compressed to	0.70"
		returned to	.74"	returned to	.82"	returned to	.94"
		springback	.04"	springback	.12"	springback	.24"
4.	70 Kaowool	free standing	1.40"	free standing	2.90"	free standing	1.30"
	30 Mullite	compressed to	0.70"	compressed to	.70"	compressed to	.70"
		returned to	0.70"	returned to	.78"	returned to	.81"
		springback	0.00"	springback	.08"	springback	.11"
5.	90 Kaowool	free standing	1.45"	free standing	2.20"	free standing	1.40"
	10 Mullite	compressed to	.70"	compressed to	.70"	compressed to	.70"
		returned to	.70"	returned to	.76"	returned to	.79"
		springback	.00"	springback	.06"	springback	.09"

5. CONCLUSIONS

The following conclusions were derived from the processes and products evaluated in this program.

1. Ceramic fibers with a high Young's modulus, such as mullite, cannot be processed on conventional textile equipment. The mechanical handling and abrasive damage causes extreme fiber damage and prevents a usable product being formed.
2. Blended felts of ceramic fibers with dissimilar mechanical properties can be readily felted on a Rotoformer. This wet-laying technique has the capacity for high production rates and the formation of a uniform product.
3. Blends of mullite fiber with Kaowool ceramic fiber produce an insulating product with good integrity and thermal stability at temperatures to 1371 C. A minimum of 30 percent mullite fiber is required for refractory purposes.
4. The addition of mullite fiber in the amounts of 30 to 55 percent improve the refractory properties of Kaowool fiber insulation and increase its resiliency. The thermal conductivity is not significantly affected by additions of mullite.

6. RECOMMENDATIONS

Additional investigation of the properties of fiber blended products should be initiated. In particular, a blend of mullite fiber with Kaowool and the addition of a ceramic binder should produce an insulating product with service temperatures in excess of 1371 C. The ceramic binder will help produce a semi-rigid board or block type insulation and will overcome some of the handling deficiencies in the blended felts. A binder could be introduced into the slurry which is fed into the Rotoformer.

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APPENDIX I

UNSUCCESSFUL TEXTILE FABRICATION PROCESSES

Those fabrication processes common to the textile industry which originally appeared to offer the best prospects for combining the mullite fiber into yarn were evaluated. The processes are briefly outlined below.

Shirley Method

This technique involves processing staple fiber into yarn by a modified carding system. The advantages of this method are that a small fiber sample can be employed and that the carding action can be slowed down to minimize damage to the fibers. A tough organic fiber may be added in this process. The organic material would be removed by dissolution or burning prior to placing the yarn in the mullite tile.

Paper Felting

Fibers are first dispersed in a water slurry and then are felted on a screen by evacuating the water. A thin layer of interlocked fiber is produced and is generally referred to as fiber paper. Figure A-1 shows a single sheet paper maker.

Scavenger Yarns

This method of processing brittle fibers into yarn involves blending in a tougher or more extensible fiber. For example, mullite could be blended with cotton or nylon in a 50:50 ratio to form a sliver. Several slivers could then be combined with the necessary twist to form the yarn.

Mechanical Twist

This technique involves gathering together sufficient mullite fiber to make the 2.48 gpd yarn. The twist necessary to form the yarn will be introduced by a standard twist tester, or any device for securing one end of a fiber bundle while the other end is rotated.

"Tow-to-Top" Method

Parallel bundles of monofilament fibers in tow form can be directly converted to yarn by converting the tow into a long staple (5-6 in.) in a manner analogous to the wool worsted top system.

Based upon the results of these techniques, alternate methods were suggested. The production of a webbing appeared feasible on a Rando-Webber. This equipment takes ceramic fiber, distributes it into a three-dimensional network and produces a thin webbing. The Webber tends to significantly reduce fiber length by the action of high speed "combing". The Rando-Webber is shown in Figure A-2.

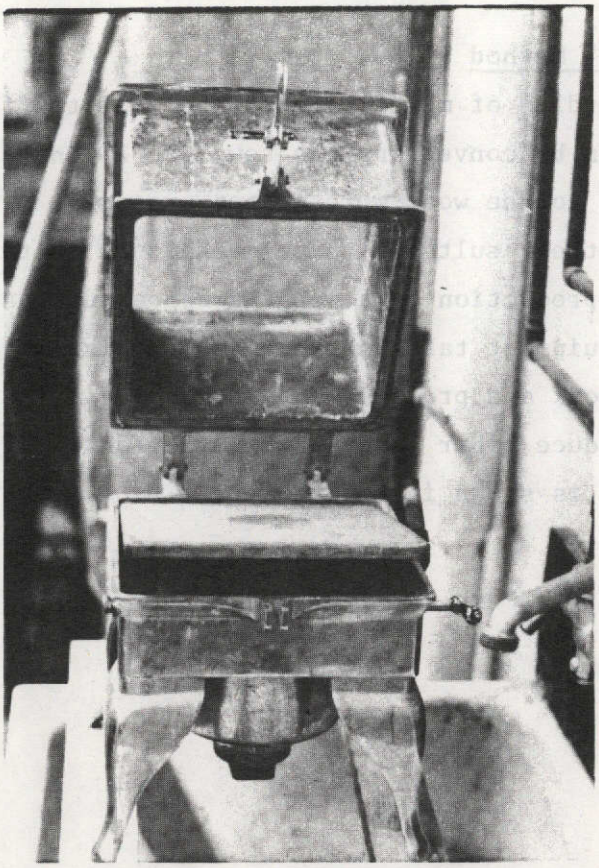

FIGURE A-1. Single Sheet Paper Maker

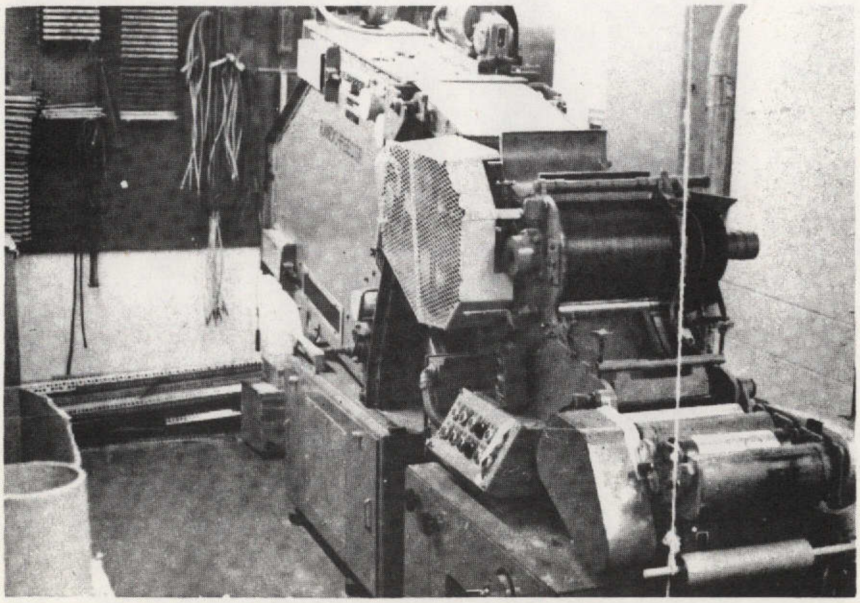

FIGURE A-2. Rando-Webber

APPENDIX II

UNSUCCESSFUL TEXTILE PROCESSING RESULTS AND DISCUSSION

1. Multifilament Tow

Continuous mullite fiber was extruded, collected, dried, and fired to 1065 C. The fired fiber exhibited good flexibility and allowed the incorporation of three tows into a loosely braided yarn.

Fiber damage due to abrasion was excessive in handling the fiber tows. Several sizings and methods of application were suggested by Fabric Research Laboratory personnel to provide a lubricated surface on the individual fibers.

Two candidate sizings which could be applied following extrusion were identified. In one case MgO is formed on the fiber surface and in the other case an aluminum chelate is formed. Two possible ways are seen as possibilities for the MgO. The first is to add magnesium acetate to the salt decomposition solution. This material is an excellent grain-growth inhibitor and is thus expected to improve the high temperature strength of the fibers. It is not known if the MgO will migrate to the surface if applied in this manner. The other technique is to first apply a starch-oil size to the fired fibers (standard in the glass industry) and then the magnesium acetate solution. Heating will then convert the magnesium acetate to magnesium oxide. This technique and the aluminum chelate technique are detailed in an AFML report on work done in the early 1960's at Fabric Research Laboratories (ASD-TDR-63-802).

The aluminum chelate size is formed by passing the fired fiber through two consecutive baths, the first containing aluminum isopropoxide in toluene and the second containing 8-hydroxy quinoline in toluene. After drying at 315 C for fifteen minutes the aluminum chelate is formed on the fiber surface. The aluminum chelate size was found to have both better anti-abrasion properties and higher temperature stability in a study on glass fibers.

Polyvinyl alcohol (PVA) was suggested to be incorporated into the fiber composition prior to extrusion. PVA typically migrates to the surface of fibers extruded in this manner, and acts as a lubricant during handling, drying, and in firing until the PVA is driven off. The addition of PVA improved the handleability of the fibers somewhat.

In addition to reducing the handling damage between monofilaments, the concept of using multifilament fiber tows to produce a yarn also depends upon the ability to increase the production level of the extruded fiber. Several improvements in the extrusion process were made to increase the production rate, including successful extrusion in a 10 orifice spinnerette. The conditions established for multi-orifice extrusion of the mullite fiber solution are given below.

a. Each orifice must be absolutely clean and circular. Spinnerettes are ultrasonically cleaned in acetic acid prior to each use. They are then examined under a microscope to determine if any circular orifice is plugged, since plugged orifices will not allow fiberization upon extrusion.

b. The spinnerette surface is coated with a spray-on fluorocarbon mold release agent. This prevents the salt decomposition solution from wetting the spinnerette surface and thus inhibits glob formation and promotes fiberization.

The requirements for and limitations to scaling up to 50-100 orifice simultaneous extrusion were investigated. There is a minimum spacing allowable between orifices on a spinnerette, below which product from two or more orifices will interact. There is also a limiting maximum 3-4 inch diameter for a spinnerette. This limit is defined by the extrusion pressure. If the diameter is too large, two problems occur. The pressure will become uneven across the face of the spinnerette, and the face of the spinnerette will deform as a result of the pressure. The optimum configuration on a spinnerette is one ring of holes around the periphery of the spinnerette. These factors lead to a maximum of about 60 orifices on a single workable spinnerette for this project.

Even with this level of scale-up, the cost of producing multifilament still far exceeds the cost of producing staple fiber. Because of this and parallel success in forming a felt with the staple fiber, the decision to scale-up was deferred.

2. Ceramic Fiber Paper

By first forming a thin fiber paper, it was planned that a yarn-like product could be produced by braiding or twisting narrow strips of the fiber paper. A single sheet paper making apparatus, as shown in Figure A-1, was utilized to evaluate this approach.

Several test sheets of fiber paper were produced at Fabric Research Laboratories from (a) staple mullite, (b) mullite with silica binder, (c) mullite with Kaowool, (d) mullite, Kaowool, and silica binder, and (e) mullite, Kaowool and latex binder.

The mullite fiber lacked structural integrity in the dried paper so the Kaowool and binders listed above were added. The Kaowool, silica, and latex all contributed significantly to improving the strength of the paper. Table VIII lists the complete range of compositions studied. Sample 10 with latex added had excellent flexibility for twisting and could be bent 180 degrees around a mandrel and resisted tearing during handling. Those compositions with colloidal silica tended to become semi-rigid.

The paper making process depends upon a binder such as latex to impart handleability. This binder would require an additional burnout step in production of a rigidized tile. The paper is not an oriented product and the production of a yarn with directional properties did not appear feasible. For these reasons, no further development or orientation of the fiber paper was performed.

3. Shirley Method

The feasibility of forming yarn on a Shirley apparatus was determined at South Eastern Massachusetts University. The Shirley system allows carding of fibers at a controlled rate, and it was planned to operate the system very slowly to minimize damage to the mullite.

Results were negative, with the mullite fiber being completely pulverized by feed rollers. Incorporation of a nylon fiber to form a scavenger yarn were also unsuccessful, with the mullite fiber being crushed before reaching the carding mechanism. Since the Shirley Method was judged to be the least destructive of all available textile fabrication methods, the remaining technique of top-to-tow was not evaluated.

Table VIII. Content of Paper Samples

Sample No.	Inorganic Fiber	Organic Carrier Fiber and Any Binder
1	10 gm Kaowool	No binder
2	15 gm Kaowool	65 ml colloidal silica (Syton DS-40)
3	10 gm Kaowool 10 gm Mullite	60 ml colloidal silica
4	6 gm Kaowool 6 gm Mullite	100 ml colloidal silica
5	8 gm Kaowool 8 gm Mullite	100 ml colloidal silica
6	8 gm Kaowool 8 gm Mullite	350 ml water glass
7	16 gm Mullite	350 ml water glass
8	8 gm Kaowool 8 gm Mullite	875 ml water glass
9	8 gm Kaowool 8 gm Mullite	21 gm latex Pressed when dry
10	8 gm Kaowool 8 gm Mullite	52 gm latex Pressed when dry
11	5 gm Kaowool 5 gm Mullite	5 gm rayon (7mm x 1.5 dpf) 20 gm latex
11(a)	Sample #11 afer 4 hours @ 500°F (260°C) to carbonize rayon	
12	5 gm Kaowool 5 gm Mullite	5 gm polyester (approximately 1/2 in. long) 20 gm latex
13	4 gm Kaowool 4 gm Mullite	20 gm latex

4. Rando-Webber Felt

Tests performed at Fabric Research Laboratories (FRL) indicated that a usable felt product could be prepared using a Rando-Webber. This equipment combs the fiber and produces a non-woven webbing or felt.

The original test runs were made using various blends of polyester fiber, 1/2 inch in length, as a carrier fiber for the less flexible mullite. Webbing was produced at 90% mullite - 10% polyester and at 80% mullite - 20% polyester blends. The webbing, or felt, was 12 inches wide and from 3/4 to 1-1/2 inches in thickness. The felts produced were sprayed with an acrylic finish to increase their handleability. The felt as it was received from FRL is shown in Figures A-3 and A-4. This felt could be handled wihtout damaging the material.

To convert the felt to a rigidized product, a series of steps was necessary to remove the organics (polyester and acrylic) and to introduce a ceramic binder. The steps which were experimentally developed were as follows:

a. Cut webbing to desired size.
b. Spray colloidal silica on surfaces and edges of cut pieces (then stack, if a multi-layered piece is to be produced).
c. Dry in air at 107 C (with flat weight on top of webbing to prevent warpage) for 8 or more hours.
d. Heat pads to 315 C for 3 hours (weighted again) to partially degrade organic material.
e. Immerse pad in colloidal silica.
f. Dry again as in (c)
g. Heat pad to 648 C at 110 C/hour (burns out remaining organic material).
h. Continue heating to 1259 C at a rapid rate (about 220 C/hour), leave one hour, cool down in furnace.
i. Remove from furnace, remove weights and proceed with further testing.

The final density of material processed in this manner is 10-15 lb/cu ft.

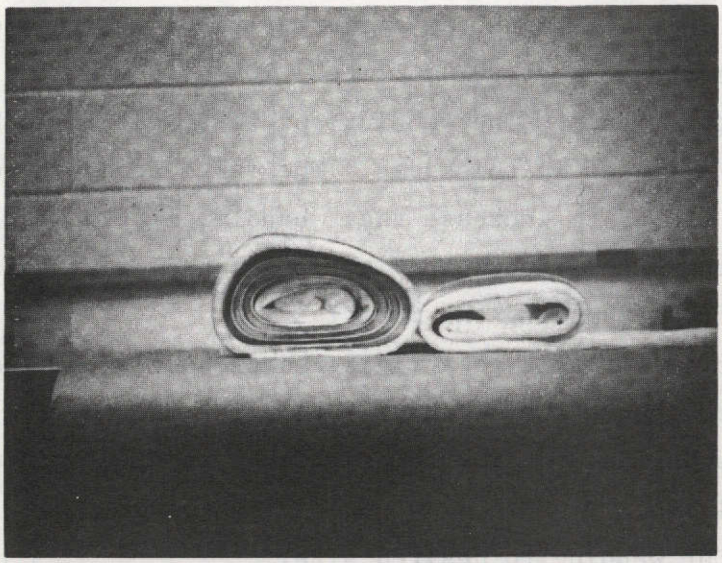

FIGURE A-3. MULLITE FIBER WEBBING AS-RECEIVED

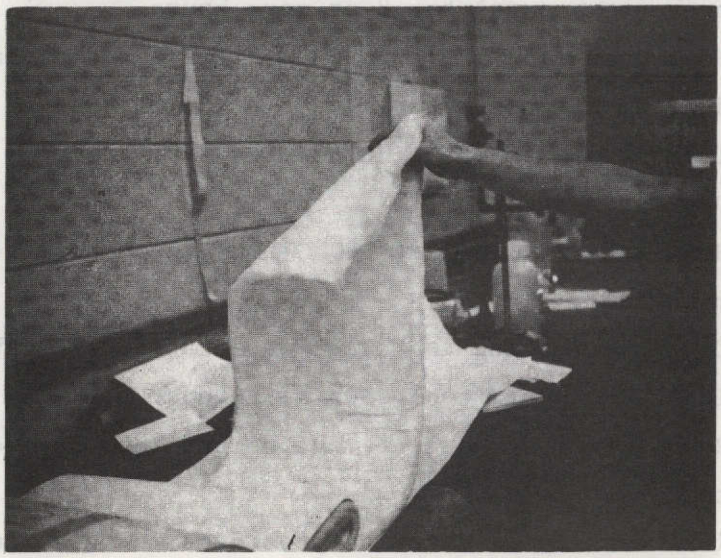

FIGURE A-4. 90% MULLITE/10% POLYESTER WEBBING
NOTE: STRUCTURAL INTEGRITY AND FLEXIBILITY

This process was used on samples of both 80% and 90% mullite. The 80% mullite lacked in structural integrity after processing, presumably due to lack of sufficient mullite fiber density in the final form. The 90% mullite material was much easier to handle and appeared to have reasonable strength.

To evaluate the tiles formed by laying up 6 plys of mullite felt, four tiles were fabricated, dried, fired to 1259 C and then measured prior to commencing thermal cycling. Ten cycles were performed to 1259 C these four tiles. The tiles were introduced into the furnace at 1259 C, soaked for one hour, and then removed and allowed to cool in air. This procedure was repeated ten times. The shrinkages on these four tiles are given in Table IX.

Table IX. Tile Shrinkage After 10 Cycles to 1259 C

	Percent Shrinkage			
	No. 1	No. 2	No. 3	No. 4
Length	0.46	0.36	(+) 0.98	1.0
Width	(+) 0.35	1.4	0.10	1.1
Height	1.5	0.59	1.9	(+) 2.2

NOTE: (+) Indicates expansion rather than shrinkage.

These tiles appear to have sufficient integrity for incorporation into an insulation system. The randomness of the fiber in the felt produced in the Rando-Webber may overcome some of the anisotropy evident in the other fiber tiles produced.

Kaowool-Mullite Blends

Based upon the progress made with adding a flexible fiber such as polyester to mullite, a series of blends of mullite combined with B&W's Kaowool alumina-silica insulating fiber were fabricated on the Rando-Webber. Six separate blended compositions ranging from 10 mullite - 90 Kaowool to 90 mullite - 10 Kaowool were fed through the Rando-Webber and very little success was attained with any of the compositions. The severe action of the high speed steel teeth in the final combing step reduced the length of the Kaowool fiber as well as the mullite fiber such that very little strength or integrity was obtained in the felted products generated. Testing of these felts for strength, shrinkage, resiliency, and handleability would have been meaningless, since the blended felts could not have been handled for installation in a refractory application at this point. The Rando-Webber was considered to be impracticable, since the addition of at least 10 weight percent of an organic carrier was required to provide handleability.