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(NASA-CR-166578) OPACIFICATION OF HIGH TEMPERATURE FIBROUS INSULATION (Manville Service Corp.) 30 p HC A03/MF A01 CSCL 11D

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Opacification of High Temperature Fibrous Insulation

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Opacification of High Temperature Fibrous Insulation

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INTRODUCTION

Background

Quilted fiber felt is currently being used as a reusable surface insulation to provide thermal protection to low thermal profile areas of the Space Shuttle Orbitor. The product consists of a silica fiber felt approximately 12mm in thickness sewn between a layer of quartz cloth on the hot face and glass cloth on the cold face. The silica fiber felts are formed by gravity and vacuum draining of a suspension of silica fibers in water. They are not bonded with organic or inorganic binder, relying totally on fiber entanglement for strength and handleability. Silica fiber felts act as thermal insulators primarily by blocking the direct path of radiation, which is by far the largest component of heat transfer through low density fibrous bodies at elevated temperatures.

There is ample evidence that the addition of strongly infrared absorbing particles such as metal oxides and carbides (opacifying particles) can reduce the overall thermal conductance of fibrous bodies. Recent in-house work with additions of silicon carbide particles to vacuum formed refractory fiber bodies resulted in reduced thermal conductivity at equivalent bulk densities. This concept of increased opacity should be directly applicable to silica fiber felts as well.

Research Objective

It was the objective of this program to study the thermal effects of addition of opacifying particles to the silica fiber felts. The addition of these particles was expected to increase both the radiation scattering and the absorption and re-emittance of energy thus reducing the total heat transfer through the felt. A second objective of the program was to provide NASA with both small and full size quilted samples of opacified felt for vibration and other thermal testing.

EXPERIMENTAL PROGRAM

Opacifiers

The particles selected for the opacification study were chromium oxide, silicon carbide and titanium dioxide. These were chosen based upon their use in other systems as opacifiers and emmittance controlling agents. Chromium oxide was used successfully in the initial coatings on LI 1500 as an emmittance controlling agent. Later, silicon carbide particles were incorporated into the LI 1500 coating to perform a similar function.

Besides the recent in-house results on silicon carbide opacification of refractory fiber felts shown in Figure 1, Stromburg and Dotts[1], U. S. Patent 4,017,404, and German Patent Application DE2940230AL all suggest SiC as an effective absorbing material for radiant energy in the 1 to 50 micron wavelength range. Titanium dioxide has been used as an opacifier in the Manville MIN-K insulation for many years due to its ability to absorb radiant energy.

The selection of these three particles was then based upon their proven ability to opacify, their non-reactivity with silica fibers at elevated temperatures, and their commercial availability in the 1-10 micron particle range. Table 1 gives a description of the three opacifying particles used in this study.

In the case of all three particles, they were designated as -325 grade suggesting less than 1 percent by weight would be larger than 44 um. The average particle size given in Table 1 reflects the average stated by the manufacturer based upon optical determination. The silicon carbide dust collector fines were analyzed for particle size distribution by the Manville optical laboratory as part of an earlier program and found to have the distribution shown in Figure 2.

There was some question early in the program as to whether these opacifiers would be retained in the silica felt during wet felting. Using a small 100 mm diameter plexiglas laboratory felting apparatus, slurries of Q-Fiber and the three opacifying particles were filtered and the filtrate checked visually for clarity. In each case, there was significant opacifier loss in the effluent from the filtration. It was immediately obvious that some form of electrochemical technique (flocculation) would be required to attach the particles to the fibers in the slurry.

FIGURE 1

APPARENT THERMAL CONDUCTIVITY OF CERAFIBER AND

5% SiC OPACIFIED CERAFIBER FELTS

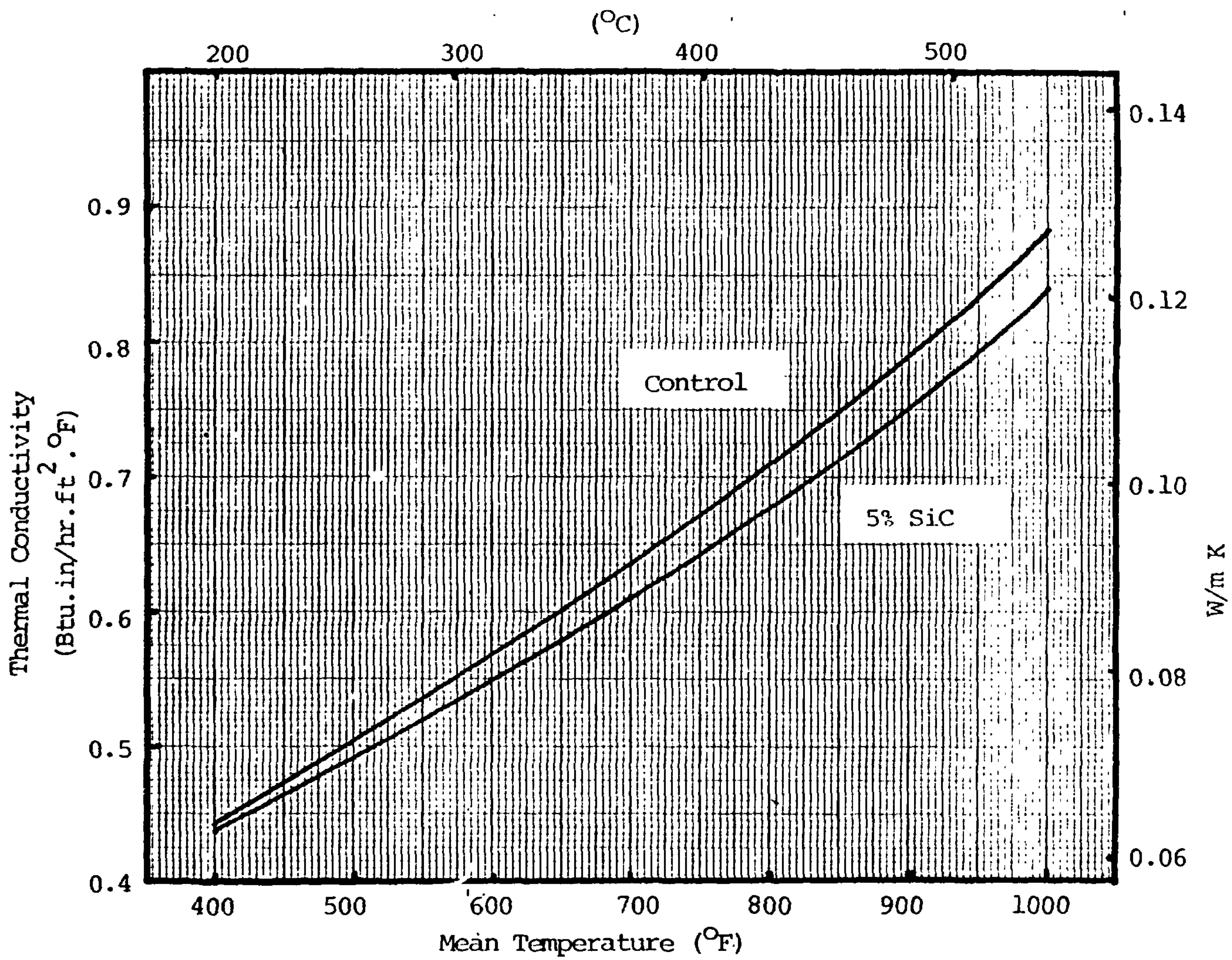


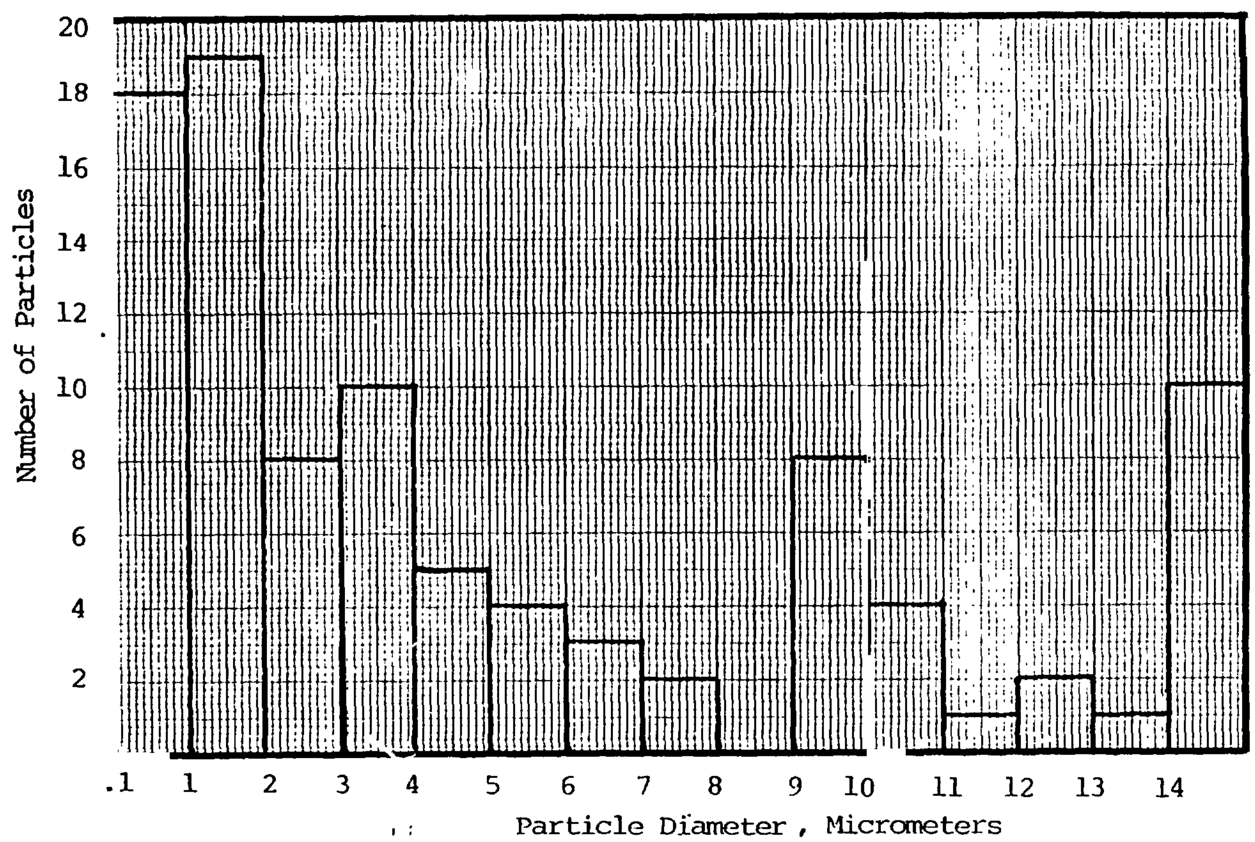
TABLE 1

Opacifying Particles

Particle	Specific Gravity	Average Particle Size	Grade Manufacturer
Cr_2O_3	5.21	4 um	-325 Cr_2O_3 3M Company Copley, Ohio
SiC	3.17	3 um	SiC Dust Collector Fines Exolon Company Tonawanda, New York
TiO_2	4.26	5 um	Heavy Grade TiO_2 National Lead Co. Niagra Falls, N. Y.

FIGURE 2

PARTICLE SIZE DISTRIBUTION OF SiC PARTICLES



At that point, a number of flocculating agents, anionic and cationic organic polymers, were tested for their ability to retain the opacifying particles. None of the polymers tested were completely effective in retaining the particles as evidenced by varying degrees of cloudiness in the effluent. Without a clear effluent there was no confidence the opacified felts would contain the calculated percentage of opacifier on a dry basis. Thus it was considered essential to flocculate the system to obtain clear effluent. One technique known to work well for retaining many types of particles in fiber slurries is the use of negatively charged colloidal silica and cationic starch. After some experimentation on slurries containing 4.25 g fiber and 0.75 g Cr_2O_3 it was determined that the addition of 12 mil Nyacol 215 colloidal silica and 3 mil of 1.0 percent solution of Sta-lok 445 cationic starch resulted in a completely clear effluent upon filtration. The Nyacol 215 colloidal silica is a product of Philadelphia Quartz having a 15 percent solids content of 4 um silica particles. The Sta-lok 445 starch is a precooked cationic starch manufactured by A. E. Staley Manufacturing Company which dissolves readily in luke warm water.

It would have been desirable not to add anything other than opacifying particles in making the experimental felts. However, it was considered necessary to retain the opacifier so that meaningful comparisons of thermal data could be made after the thermal tests were completed. It was at that point that the decision was made to flocculate all slurries with 3.6 percent silica and 0.6 percent starch based upon the dry weight of the fiber or the fiber and opacifier. Thus, all fiber control samples used in thermal testing also contained the flocculating agents used in the opacified felts. During the production of QUILITE blankets, the silica fiber felts are put through a 500°C heat treatment. Since this heat treatment would burn away the small amount of starch present in the felt, all experimental felts were heated in air to 500°C for a minimum of two hours prior to thermal testing or being sent to the Manville Plant for sewing into blankets.

Laboratory Felt Preparation

With the parameters for felt production established, all silica fiber control felts and silica fiber opacified felts for this study were produced using a small felting tower. This piece of equipment produced felts measuring 356 mm by

356 mm and having a thickness dependent upon the slurry input. The dam above the screen surface which was used to contain the slurry during gravity dewatering was 305 mm tall allowing for a slurry volume of approximately 40 l.

The fiber used in all felts was standard Manville Q-FIBER supplied by our Waterville plant in the same form as used in the production of Q-FIBER felt for QUILITE. In the production of the 12 mm and 30 mm thick felts for this program, the fiber was dispersed in approximately 40 l of tap water using a triple blade propeller-type mixer operating at approximately 1200 rpm. The mixing procedure involved dispersing the fiber and opacifier by mixing for five minutes. The colloidal silica was then added and the slurry mixed 1 minute at which time the starch was added. The slurry was then mixed slowly with a large spatula for approximately 1 minute. The slurry was then poured into the felting tower.

The composition of the first set of experimental felts to be made is given in Table 2 along with their physical properties after drying and trimming. Felts 1041-34-4-1...9 were made and sent to the Manville Plant for sewing into blankets for subsequent NASA vibration testing. During the sewing operation considerable difficulty was encountered in stitching the opacified felts. These problems were attributed to unfamiliarity with sewing a new and somewhat different type of felt. Also it was realized that due to miscommunication, the felts were not heat treated prior to stitching and it was thought the presence of starch might have caused some of the difficulties. Several of the felts containing 15 percent Cr_2O_3 were unstitched, heat treated and then resewn. There was some improvement in the ability to sew the felts which was in part due to increased experience with the material. Chrome oxide was chosen for the initial vibration testing because its distinct color would facilitate visual evaluation of any migration of opacifier within the felt, or through the stitch holes and cloth to ambient.

The subsequent sample series (felts 1041-34-5-1...4) were designed to have an unfinished thickness of 32 mm, assuming 96 kg/m^3 density, and were prepared for ASTM C-518 thermal conductivity tests. After drying these four felts,one control and one each containing 10 percent of each opacifier were heat treated at 500°C for two hours and then trimmed to 305 mm by 305 mm. The final preparation was done on a horizontal band saw trimming the faces of the samples to obtain flat parallel surfaces. To achieve constant density,

TABLE 2

Comparison and Physical Properties of Initial Cr_2O_3

Opacified Felts for Vibration Testing

Felt ID	Opacifier	Thickness mm	Density kg/m³	(lb/ft³)
1041-34-4-1	None	15.2	85	5.3
1041-34-4-2	None	14.7	92	5.8
1041-34-4-3	None	14.1	95	6.0
1041-34-4-4	15% Cr_2O_3	11.4	120	7.5
1041-34-4-5	15% Cr_2O_3	11.4	121	7.6
1041-34-4-6	15% Cr_2O_3	10.8	123	7.7
1041-34-4-7	15% Cr_2O_3	11.7	117	7.3
1041-34-4-8	5% Cr_2O_3	12.4	104	6.5
1041-34-4-9	5% Cr_2O_3	12.8	108	6.7

the sample having the highest apparent density after edge trimming, 1041-34-5-1, was face trimmed until flat surfaces were achieved. The final thickness was 22.4 mm and weight was 258 grams. The other three felts were then trimmed until their face surfaces were flat and their weights were 258 grams ± 1 gram. These samples were then tested in the high temperature heat meter operating at 427°C (800°F) at a thickness of 22.1 mm insuring equal density. The data from these thermal conductivity tests are given in Table 3.

Due to the difficulties encountered with sewing the first felts at Manville, a second series of 12 mm by 305 mm by 305 mm felts were made. Table 4 gives the composition and physical properties of these felts. After fabrication and edge trimming, these were heat treated for three hours at 500°C and then sent to Manville for application of the quartz cloth and glass cloth to the surfaces. The felts were sewn between layers of J. P. Stevens 570 Astroquartz cloth and J. P. Stevens 85392/38/9383 S Glass cloth using Astroquartz Q-24 thread, also a product of J. P. Stevens.

With the starch burned out of these felts, they were not as difficult to stitch as the first set. In order to assess the effects of the colloidal silica and Cr_2O_3 on stitching, felt 1041-34-8-10 was made with fiber only. Felt 1041-34-8-9 was made without Cr_2O_3 but with the starch and Nyacol 215 just as if it contained opacifier. Felts 1041-34-8-1...8 all contained 5 percent Cr_2O_3. There seemed to be no difference in the stitching of the two felts without Cr_2O_3. With 5 percent Cr_2O_3 about 30 percent of the stitches were not acceptable after the first pass through the multi-needle sewing equipment. It was observed that the difficulty was confined to certain needles. It was necessary to remove some of the needles from the machine and offset the panels for resewing to repair the defective stitch lines. The final closeout stitch was performed using a single needle machine with little difficulty.

It was the observation of Manville personnel that the presence of the Cr_2O_3 particles offered increased abrasion and resistance to needle penetration but with more experience, sewing these felts could be accomplished without major difficulties. These ten felts were marked 1 through 10 corresponding to the last number in their research designation and sent to NASA for vibration testing.

Based upon the results of the C-518 thermal conductivity tests on felts containing 10 weight percent of the three opacifiers, TiO_2 appeared to have the greatest effect in

TABLE 3

ASTM C-518 Tests of 10% Opacified

Silica Fiber Felts at 427°C

Felt ID	Opacifier	Density kg/m^3	Density (lb/ft^3)	Apparent Thermal Conductivity W/m k	Apparent Thermal Conductivity $(Btu.in/hr.ft^2.°F)$	k_a x Density
1041-34-5-1	Cr_2O_3	122.4	7.65	0.0776	0.539	9.49
1041-34-5-2	SiC	122.1	7.63	0.0766	0.532	9.35
1041-34-5-3	TiO_2	122.4	7.65	0.0753	0.522	9.22
1041-34-5-4	None	121.9	7.61	0.0780	0.542	9.50

TABLE 4

Composition and Physical Properties of Final Cr_2O_3 Opacified Felts for Vibration Testing

Felt ID	Opacification	Thickness (mm)	Density kg/m^3	(lb/ft^3)
1041-34-8-1	5% Cr_2O_3	12.4	107	6.7
1041-34-8-2	5% Cr_2O_3	12.6	106	6.6
1041-34-8-3	5% Cr_2O_3	12.1	107	6.7
1041-34-8-4	5% Cr_2O_3	12.4	107	6.7
1041-34-8-5	5% Cr_2O_3	12.6	106	6.6
1041-34-8-6	5% Cr_2O_3	12.6	106	6.6
1041-34-8-7	5% Cr_2O_3	12.6	106	6.6
1041-34-8-8	5% Cr_2O_3	12.6	106	6.6
1041-34-8-9	None	12.4	102	6.4
1041-34-8-10	None	16.0	85	5.3

reducing k_a at 427°C (800°F). Since the differences between conductivities of the 10 percent opacified felts was small, it was decided to make the test felts containing five weight percent addition of all three opacifiers and another control sample. Table 5 gives the initial composition and final thicknesses and densities of these felts after trimming to 305 mm by 305 mm. Table 6 gives the results of the 427°C ASTM C-518 tests of these felts compressed to 22.1 mm.

At 5 weight percent loading, only TiO_2 resulted in a k_a value lower than the control sample without opacifier. In the 10 percent opacified series the lowest k_a and k_a x density values were also found in the TiO_2 opacified sample. However, for the sake of comparison, the control sample in each experiment was compressed from approximately 96 kg/m^3 to the density of the opacified samples.

To gain a more meaningful comparison of opacified felt to felt containing only fiber, the control for the 15 weight percent TiO_2 test was changed. For this test, one sample was made containing 348g of Q-fiber and 61.5 g TiO_2. The control sample was made containing 348 g Q-Fiber. Both were made with colloidal silica and starch and then heat treated two hours at 500°C after drying. In the preparation of previous samples, each sample of the series was trimmed until constant weight was achieved. They were then compressed to the thickness of the thinnest sample in the C-518 heat meter thus achieving constant density. In the preparation of these two samples after edge trimming to 305 mm by 305 mm, both were then face trimmed until 22.3 mm thickness was achieved. They were then tested per ASTM C-518 at 427°C at 22.2 mm thickness like the previous samples. The results of these tests are given in Table 7.

Two conclusions were drawn from analysis of the C-518 apparent thermal conductivity data. At 5 and 10 percent loading only TiO_2, as an opacifying agent, resulted in consistently lower k_a values than control felts at the same density. However, comparing the product of density times thermal conductivity suggested that the reduction in k_a due to addition of TiO_2 does not offset the increase in density resulting from that addition.

Up to this point, all thermal conductivity tests were conducted at 427°C. It is well known that at higher temperatures, the radiation component represents a much larger portion of the total heat transfer. For this reason the addition of an opacifying particle would be expected to

TABLE 5

Composition and Physical Properties

of 5% Opacified Felts for ASTM C-518 Tests

Felt ID	Opacifier (type)	Opacifier (g)	Fiber (g)	Final Thickness (mm)	Final Weight (g)
1041-34-10-1	Cr_2O_3	19.1	363	22.6	230.7
1041-34-10-2	SiC	19.1	363	22.5	230.3
1041-34-10-3	TiO_2	19.1	363	22.6	230.9
1041-34-10-4	None		382	23.9	230.6

TABLE 6

ASTM C-518 Tests of 5% Opacified

Silica Fiber Felts at 427°C

Felt ID	Opacifier	Density kg/m^3	Density (lb/ft^3)	Apparent Thermal Conductivity W/m K	Apparent Thermal Conductivity (Btu.in/hr.ft^2.°F)	k_a x Density
1041-34-10-1	Cr_2O_3	108.6	6.79	0.0792	0.550	8.60
1041-34-10-2	SiC	108.3	6.77	0.0795	0.552	8.61
1041-34-10-3	TiO_2	109.1	6.82	0.0782	0.543	8.53
1041-34-10-4	None	109.1	6.82	0.0787	0.547	8.59

TABLE 7

ASTM C-518 Tests of 15% TiO_2 Opacified

Silica Fiber Felt at 427°C

Felt ID	Opacifier	Density kg/m^3	Density (lb/ft^3)	Apparent Thermal Conductivity W/m K	Apparent Thermal Conductivity (Btu.in/hr.ft^2.°F)	k_a x Density
1041-34-11-1	TiO_2	116.3	7.27	0.0750	0.521	8.72
1041-34-11-2	None	93.9	5.87	0.0828	0.575	7.77

be more effective at reducing thermal conductivity at higher mean temperatures. To measure the effect of 10 weight percent TiO_2 loading on Q-Fiber felt at mean temperatures up to 900°C a set of samples were made for ASTM C-201 testing.

The ASTM C-201 calorimeter requires two 25 mm thick layers of felt 457 mm long by 305 mm wide. Consequently, four 305 mm by 305 mm by 25 mm felts containing 10 weight percent TiO_2 and four felts of the same size containing only fiber were made and heat treated. From the four felts of each type, sufficient material was cut and pieced together such that no joint existed in either the top or bottom layer above the 76 mm by 76 mm metering area centrally located in the calorimeter.

The apparent thermal conductivity tests were conducted on the opacified and control samples per ASTM C-201 between mean temperatures of 100°C and 950°C to obtain the curves given in Figure 3. Again, the apparent thermal conductivity of the TiO_2 opacified sample was lower than that of the control. In this case, the densities of the two materials, 100.8 kg/m^3 and 92.8 kg/m^3 for 10 percent TiO_2 and control felts respectively, were not as far apart as in the previous tests. To test the original assumption that the effect of opacification would become more beneficial with increasing tempertaure, the percent reduction in k_a between the opacified felt and the control felt was plotted against mean temperature in Figure 4. The results indicated that above 204°C, the effect of TiO_2 in reducing k_a continues to increase through 870°C.

Titanium dioxide was considered to be the most efficient opacifier, and was used exclusively in the experimental production run. However, in spite of all attempts to correct the situation, the density of the laboratory-made opacified felts were always higher than the unopacified felts, thus casting doubt on the value of the opacification.

Experimental Production Run

Arrangements were made with the Manville production facility at Waterville, Ohio to manufacture five 3 m x 1 m (nominal) felts using standard production techniques. The felts are described in Table 8.

It will be noted that two felts (5 and 6) were made by adding 10 and 15 percent TiO_2 to the standard 6 pcf felt

FIGURE 3

ASTM C-201 APPARENT THERMAL CONDUCTIVITY

Q-FELT AND Q-FELT WITH 10% TiO_2

(LABORATORY- MADE FELTS)

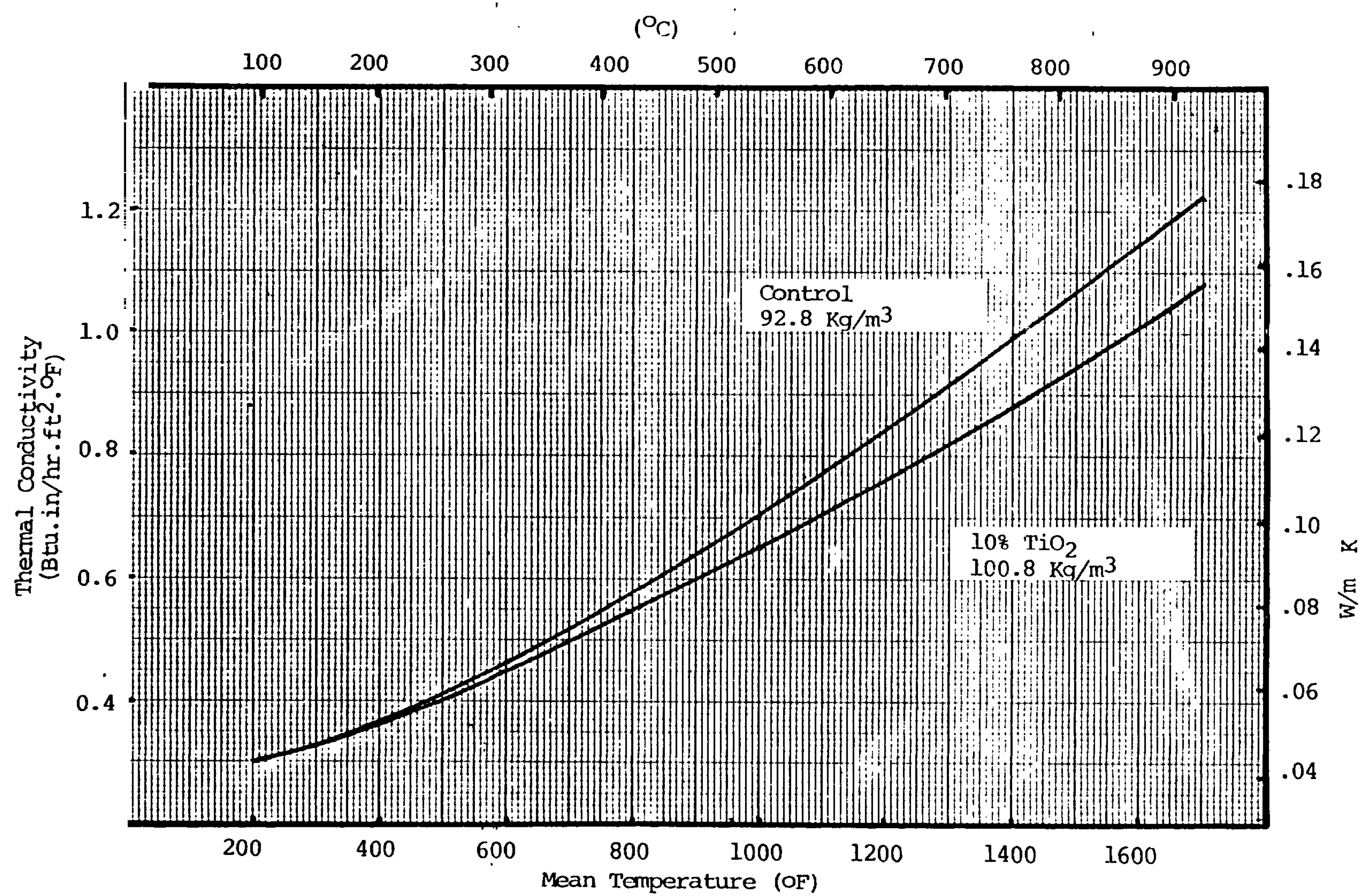


FIGURE 4

PERCENTAGE REDUCTION IN THERMAL CONDUCTIVITY

DUE TO 10% TiO_2 ADDITION

(LABORATORY-MADE FELTS)

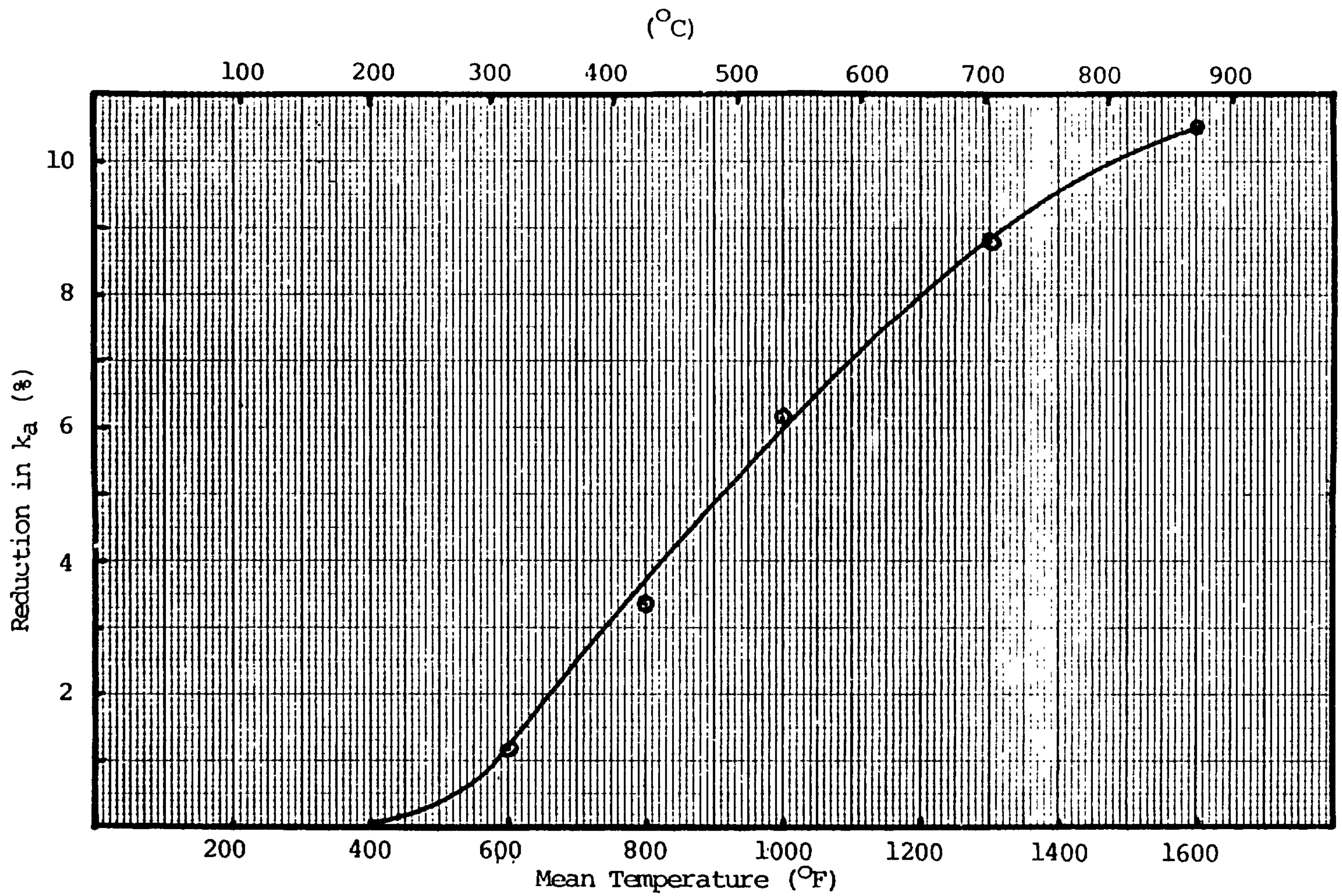


TABLE 8

Description of Experimental Production Felts Containing TiO_2 Opacifier

Felt ID	Nominal Density kg/m^3	(pcf)	Nominal Thickness mm	(inches)	TiO_2 Opacifier Content %
1041-34-16-1	96	6	12.5	0.5	0
1041-34-16-2	96	6	12.5	0.5	10.0
1041-34-17-3	96	6	12.5	0.5	15.0
1041-34-17-4	107	6.7	12.5	0.5	9.8
1041-34-17-5	114	7.1	12.5	0.5	14.8

rather than limiting the study to the substitution of TiO_2 for fiber as in felts 2 and 3. While the density was definitely increased, the intent was to confirm or reject the earlier conclusion that the product of thermal conductivity and density (kp) would not be significantly lower with the higher density felts.

In order to enhance the retention of the opacifier by the fiber matrix, 0.5% Nalco OSJ-600 flocculating agent was added (based on the total fiber and opacifier weight). Nalco OSJ-600 was found to be a satisfactory flocculating agent after the laboratory specimens had been made. This addition was made after the standard fiber/opacifier slurrying cycle, and using the mixer very sparingly to prevent breakup of the long chain flocculant molecule.

After dewatering, the felts were dried and fired in the same manner used to produce standard felts for the shuttle program.

After drying, it was noted that the felts were quite fragile. An examination of the fibers by scanning electron microscope at the Manville Research and Development Center indicated that the fibers were significantly shorter than those from the laboratory-prepared felts. Brief laboratory trials using these earlier fibers were conducted to determine if the flocculant or the opacifier were the cause of the short fibers. Intensive high speed mixing, and prolonged exposure of the raw fibers to elevated humidity (to accelerate moisture attack on the unfired leached fibers) did not result in a similar shortening of the fibers. Thus, while the cause was not clearly established, the addition of the flocculant and opacifier were tentatively ruled out.

The experimental production felts were shipped to the Manville Research and Development Center where they underwent extensive thermal conductivity evaluation by the ASTM C-201 High Temperature Calorimeter, and the ASTM C-518 Rapid Heat Meter. The results following regression analysis of the ASTM C-201 data are presented in Table 9. Table 10 presents the regression equation, and the standard deviation for each of the ASTM C-201 Calorimeter tests.

It is important to note that inaccuracies due to sample thickness measurement, and thus sample density and thermocouple placement must be expected in ASTM C-201 tests involving low density multilayer fibrous felts. ASTM C-201 was originally designed for use with rigid materials such as

TABLE 9

THERMAL CONDUCTIVITY OF EXPERIMENTAL PRODUCTION Q-FELT AND Q-FELT CONTAINING TiO_2 OPACIFIER

	1041-34-16-1		1041-34-16-2		1041-34-17-3		1041-34-17-4		1041-34-17-5	
ASTM C-201 Calorimeter										
Apparent k, W/m K (Btu in/hr ft^2°F)										
Mean Temperature: 93C (200F)	0.0396	(0.28)	0.0403	(0.28)	0.0398	(0.28)	0.0401	(0.28)	0.0420	(0.29)
204C (400F)	0.0539	(0.37)	0.0530	(0.37)	0.0506	(0.35)	0.0517	(0.36)	0.0523	(0.36)
316C (600F)	0.0697	(0.48)	0.0661	(0.46)	0.0621	(0.43)	0.0640	(0.44)	0.0628	(0.44)
427C (800F)	0.0877	(0.61)	0.0798	(0.55)	0.0743	(0.52)	0.0771	(0.54)	0.0738	(0.51)
538C (1000F)	0.1088	(0.76)	0.0945	(0.66)	0.0876	(0.61)	0.0916	(0.64)	0.0853	(0.59)
649C (1200F)	0.1337	(0.93)	0.1102	(0.77)	0.1023	(0.71)	0.1077	(0.75)	0.0975	(0.68)
760C (1400F)	0.1632	(1.13)	0.1272	(0.88)	0.1185	(0.82)	0.1256	(0.87)	0.1104	(0.77)
ASTM C-518 Rapid Heat Meter										
Apparent k, W/m K (Btu in/hr ft^2°F)										
Mean Temperature: 316C (600F)	0.0685	(0.48)	0.0611	(0.42)	0.0634	(0.44)	0.0643	(0.45)	0.0625	(0.43)
427C (800F)	0.0845	(0.59)	-	-	-	-	0.0775	(0.54)	0.0750	(0.52)
538C (1000F)	0.1041	(0.72)	0.0894	(0.62)	0.0935	(0.65)	0.0939	(0.65)	0.0900	(0.63)

TABLE 10

REGRESSION ANALYSIS* OF ASTM C-201 DATA

FOR Q-FELT AND Q-FELT CONTAINING TiO_2 OPACIFIER

Felt ID	Regression	Correlation Coefficient	Standard Deviation
1041-34-16-1	$Y = 0.1098 \times 10^{-9} X^3 + 0.4648 \times 10^{-3} X + 0.1813$	0.999	0.0095
1041-34-16-2	$Y = 0.3171 \times 10^{-10} X^3 + 0.4308 \times 10^{-3} X + 0.1936$	0.991	0.025
1041-34-17-3	$Y = 0.3885 \times 10^{-10} X^3 + 0.3673 \times 10^{-3} X + 0.2023$	0.989	0.024
1041-34-17-4	$Y = 0.4565 \times 10^{-10} X^3 + 0.3903 \times 10^{-3} X + 0.2003$	0.991	0.023
1041-34-17-5	$Y = 0.1974 \times 10^{-10} X^3 + 0.3510 \times 10^{-3} X + 0.2214$	0.971	0.035

*Where: X = Mean Temperature, oF

Y = Apparent Thermal Conductivity, Btu in/hr $ft^2 {}^oF$

** Btu in/hr ft^2 ^oF

insulating firebrick in the 2-3-inch thickness range. Since the cold face of the sample is always near room temperature, the ΔT across the sample is generally high, and the maximum mean temperature obtainable is relatively low. When testing multi-layered low density felts, it is desirable to lower the ΔT and increase the obtainable mean temperature. This is accomplished by placing thermocouples between each layer. However, as the top plate of the test unit is placed over the composite sample, it cannot be assumed that each layer is compressed exactly to the extent that the composite is compressed, thus giving rise to the potential inaccuracies of individual layer thickness, density and thermocouple location.

In this instance, the weight of the top plate would not adequately compress most of the felts to the desired nominal density. After testing, the top plate was removed, and the total thickness of the sample was measured immediately by the pin and disc method. However, some regain can be expected. Thus, the actual test density was probably somewhat higher. While this problem may appear to be of considerable magnitude, our immediate concern was the difference between samples, rather than the absolute accuracy of any one sample. In spite of these problems, the ASTM C-201 data were not significantly different from the ASTM C-518. Of greater importance however, was the fact that the 10 percent TiO_2 felts (based on substitution for fiber) could be made and tested at essentially the same nominal density as the control felt.

The regressions presented in Table 10 show very distinctly the effect of opacification. The coefficient for the cube of the temperature for the standard felt carries a 10^{-9} factor, while that for all other felts is 10^{-10}. Further, the standard felt carried the largest coefficient for the linear temperature term. Conversely, but as expected, the standard felt had the lowest apparent conductivity when "x" equaled zero. At these low temperatures, radiation is minimal, and the replacement of particles for fibers or the increase in density adversely impacts the apparent conductivity.

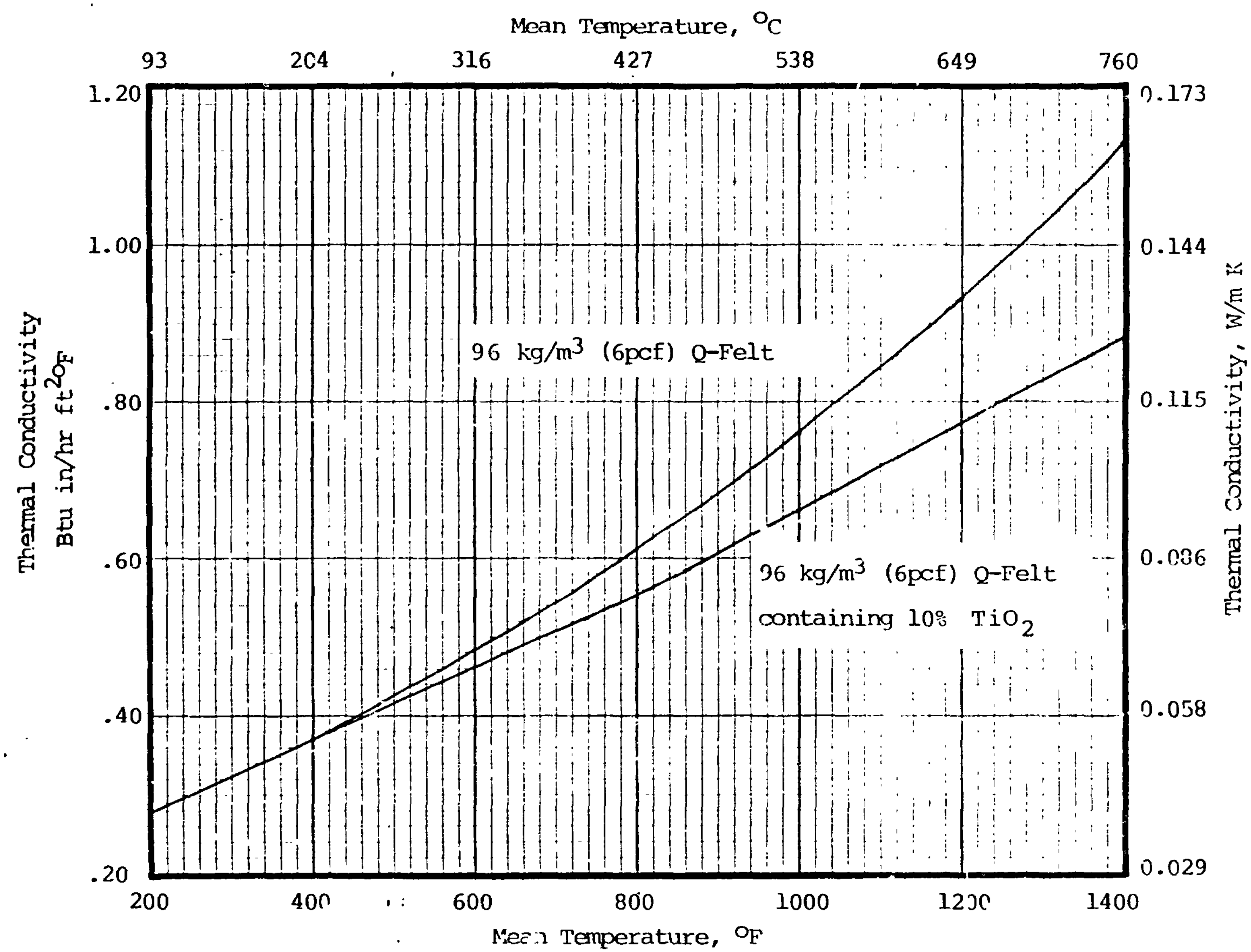
These regressions were evaluated for significant differences between them. Based on the limited amount of testing, a significant difference was found between the standard felt, and the 10 percent TiO_2 and 15 percent TiO_2 felts. However, no significant difference could be found between the two levels of opacification. The last two felts, where the opacifier was added to the 6 pcf fiber rather than substituted for an equal amount of fiber, were also significantly improved over the standard felt, but not significantly better than the other opacified fel s. Thus, the earlier work was confirmed.

Figure 5 shows a plot of apparent thermal conductivity versus mean temperature for the standard felt and the 10 percent opacified felt (1041-34-16-1 and 1041-34-16-2 respectively). These plots distinctly show the effect of the opacifier with the near straight line condition of the 10 percent TiO_2 felt.

The percent improvement of the 10 percent TiO_2 felt over the standard felt appears to range from zero at the low temperatures to slightly under 25 percent at 760C (1400F). This magnitude of improvement, if borne out by extensive repetitive testing, represents a very important achievement and a major step towards greater thermal protection, or less weight.

FIGURE 5

THERMAL CONDUCTIVITY VERSUS MEAN TEMPERATURE FOR Q-FELT AND Q-FELT CONTAINING 10% TiO_2 OPACIFIER (EXPERIMENTAL PRODUCTION FELTS)



CONCLUSIONS

Based on the above results, the following conclusions appear reasonable:

1. The addition of an opacifier to the extent of 10 to 15 percent of the total weight of a 96 kg/m^3 (6 pcf) microfiber felt reduces the thermal conductivity of the felt.

2. Titanium dioxide having an average particle size of 5 micrometers appears to perform best as an opacifier when compared with 4 micrometer Cr_2O_3 and 3 micrometer SiC.

3. The apparent thermal conductivity of a 96 kg/m^3 (6 pcf) 10 percent TiO_2 felt was significantly lower than that of the standard 96 kg/m^3 (6 pcf) felt.

4. The apparent thermal conductivity of a 96 kg/m^3 (6 pcf), 15 percent TiO_2 felt was significantly lower than that of the standard 96 kg/m^3 (6 pcf), felt, but not significantly lower than that of the 96 kg/m^3 (6 pcf) 10 percent TiO_2 felt.

5. The 10 percent and 15 percent TiO_2 additions to the 96 kg/m^3 (6 pcf) felt resulting in higher product densities were not sufficiently better than the 10 or 15 percent TiO_2 substitution for fiber to warrant the added weight.

6. Assuming the 10 percent TiO_2 opacified 96 kg/m^3 (6 pcf) felt to be the optimum, the percent improvement over the standard 96 kg/m^3 (6 pcf) felt ranged from zero at 93C (200F) and 204C (400F) mean temperatures to just under 25 percent at 760C (1400F) mean temperature.

7. The addition of the opacifier and a small amount of flocculant did not appear to affect adversely the production process.

8. The short fibers experienced during the experimental production run could not be attributed to the addition of the opacifier and flocculant based on laboratory trials.