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THERMAL INSULATIONS FOR LAUNCH VEHICLE RADIANT HEATING ENVIRONMENTS

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ABSTRACT

A ceramic composite thermal insulation was developed as an alternate material for M-31, the insulation presently used on the aft sections of the booster stages of the Saturn IB and Saturn V launch vehicles. The material, designated as FTA-442A insulation, is comprised of alumina-silica fibers, pigmentary potassium titanate, and asbestos fibers bonded with colloidal silica. The insulating efficiency of the FTA-442A insulation in both radiant and convective heating environments is superior to that of M-31. It is suitable for application to metal substrates, including stainless steel honeycomb sandwich structures, and the mechanical strength and related substrate adhesion are superior to M-31. The material development is described, and selected thermal, optical, and mechanical properties of the insulation are reported and compared to M-31.

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PROPULSION & VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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SUMMARY

A ceramic composite thermal insulation was developed as an alternate material for M-31, the insulation presently used on the aft sections of the booster stages of the Saturn-class launch vehicles. The material, designated as FTA-442A insulation, is an unfired, highly reflective ceramic coating comprised of alumina-silica fibers, pigmentary potassium titanate, and asbestos fibers bonded with colloidal silica. The material development is described, and selected thermal, optical, and mechanical properties of the insulation are reported and compared to M-31.

The insulating efficiency of FTA-442A insulation, in both radiant and convective heating environments, is superior to M-31. The insulation can be applied to metal substrates, including stainless steel honeycomb sandwich structures, and the mechanical strength and related substrate adhesion are superior to M-31.

This new insulation has uniform properties because it is not as subject to silica binder migration as the M-31 insulation.

Engineering properties of interest that were determined for FTA-442A insulation are as follows:

a. Bulk density - 61 lbs/ft^3

b. Water absorption - 44 percent

c. Modulus of rupture - 1535 psi

d. Reflectance - 83.5 percent at 1.7 microns

e. Specific heat - 0.246 Btu/lb-°F in the 25-100°C (77-212°F) temperature range

f. Thermal conductivity - 1.34 and 1.77 Btu/ft^2/hr/°F/in at 93°C (200°F) and 426°C (800°F), respectively

g. Melting point - approximately 1315°C (2400°F)

h. Good thermal-shock resistance.

INTRODUCTION

An insulating material designated M-31 was developed by Seitzinger (ref. 1 and 2) for use as a heat shield insulation on the aft sections of Saturn-class launch vehicles. M-31 is an unfired, highly reflective, inorganic material composed of Tipersul* (fibrous potassium titanate) and asbestos fibers** bonded with Ludox HS*** (colloidal silica sol). The thermal environment imposed upon the base region of the Saturn vehicles is induced by two sources; radiation from the engine plumes and convection from the recirculating exhaust gases. Nominally, 60 to 80 percent of the total heat load is radiative. Therefore, an insulation with high reflectance in the infrared region was desirable. Since fibrous potassium titanate has high infrared reflectivity, it was used as the primary ingredient of M-31.

Utilization of M-31 as a heat shield insulation requires that it be applied in thick coatings to heat shield substrates. The heat shield substrates for both the S-IB and S-IC stages are constructed of a 1-inch thick stainless steel honeycomb core brazed between thin stainless steel face sheets. A 1/4-inch thick open-faced stainless steel honeycomb reinforcement is brazed to one of the face sheets and crimped to a prescribed height. This crimped reinforcement is used to mechanically anchor the insulation to the substrate.

M-31 is an unfired ceramic material and derives its strength only from the bonding action of the colloidal silica. During the drying of M-31, the silica migrates toward the outer surface of the insulation, resulting in a material with a dense, strong outer layer and a soft, relatively weak inner layer. These conditions offer the advantages of an exterior sufficiently hard to resist erosion and physical abuse, and a lightweight interior which serves as an excellent insulator.

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*Tipersul - Trade name of E. I. du Pont de Nemours, Incorporated
**Asbestos fibers - Number 1 Chrysotile AAAA grade with a maximum iron content of 1.3 percent by weight reported as ferrous oxide (FeO), Asbestos Corporation of America, Garwood, New Jersey
***Ludox HS - Trade name of E. I. du Pont de Nemours, Incorporated

However, these same effects limit the adherence of the insulation to the heat shield substrate. This adherence is governed largely by the strength of the insulation in the layer filling the voids of the crimped open-faced honeycomb which mechanically locks the insulation to the substrate. This is the zone which is most affected by the tendency of the silica binder to migrate to the surface during drying.

Fibrous potassium titanate was an experimental material when M-31 was developed. In fact, at that time, it was being produced in small batches for evaluation only. This method of production resulted in variations in the fiber size of the fibrous potassium titanate. Later, the production process was optimized and changed from the batch type to a continuous operation. The manufacturer indicated that the continuous process would provide fibers of uniform size. However, the size of the fibers still varied from batch to batch. These variations, resulting from the manufacturer's in-process reduction of fiber size, had adverse effects on the properties of M-31 such as wet workability, resistance to drying cracks, and mechanical strength. Because of the variations in the physical properties of fibrous potassium titanate, and because M-31 has limited adherence to metal substrates, a program was undertaken to develop an alternate insulation. The alternate material was required to possess substantially greater strength and related substrate adhesion than M-31, and its insulating capabilities in both radiant and convective thermal environments must be at least equal to M-31.

The first approach was the evaluation of larger inorganic fibers as replacements for the fibrous potassium titanate. Fibers evaluated were Fiberfrax*, Refrasil**, and Micro-Quartz***. Candidate insulations were prepared with each of the above materials by substituting them for fibrous potassium titanate in the M-31 formulation. The insulating capabilities of these insulations in a radiant heating screening test were inferior to M-31. Consequently, it was decided to retain some of the fibrous potassium titanate as an insulation ingredient.

All candidate insulations bonded with Ludox HS colloidal silica had strong exteriors and soft, weak interiors, due to the same type of silica binder migration that had been observed in M-31. In an attempt to minimize this effect, other colloidal silicas were evaluated as the binder. Colloidal silicas evaluated were Nalcoag**** 1034A,

*Fiberfrax - Trade name of the Carborundum Company
**Refrasil - Trade name of H. I. Thompson Fiber Glass Company
***Micro-Quartz - Trade name of Johns-Manville Company
****Nalcoag - Trade name of Nalco Chemical Company

1050, and 1060, and Ludox HS-40. For various reasons, none of these silica products were satisfactory as a substitute for Ludox HS. Another method previously found to be effective (ref. 2) in improving the texture of M-31 is to gel the Ludox HS colloidal silica binder which retards silica migration. Ludox HS is an alkaline silica sol having a pH of 9.7. Gelation of an alkaline silica sol occurs when the negative charge on the silica particles is reduced sufficiently to overcome repulsive forces, thus allowing particle linking and a subsequent network formation. The addition of sulfuric acid accomplishes the surface charge reduction and lowers the pH to the desired level. Gel time for a sol of given silica concentration is a function of both pH and temperature, as shown in FIGS 1 and 2.

Based upon this technique, an optimum procedure was established for gelling the Ludox HS. Using this procedure , and various combinations of inorganic fibers, including fibrous potassium titanate, candidate insulations were prepared and evaluated as alternates for M-31. An alternate insulation was developed that possessed greater strength and related substrate adhesion than M-31, and its insulating capabilities in both radiant and convective heating environments were superior to M-31. It was composed of Fiberfrax fibers, fibrous potassium titanate and asbestos fibers bonded with gelled colloidal silica. This material was designated FTA-442 insulation.

Upon completing the development of FTA-442 insulation, the manufacturer of fibrous potassium titanate advised that its production would be discontinued, and that it would not be available after June 30, 1966. The manufacturer recommended a new material, pigmentary potassium titanate, as a replacement for fibrous potassium titanate. The compositions of the two materials are very similar. The primary difference is in fiber size - pigmentary potassium titanate is composed of much smaller fibers. Efforts to substitute pigmentary potassium titanate for fibrous potassium titanate in the FTA-442 formulation resulted in the development of a satisfactory alternate material for M-31. The new material has been designated as FTA-442A insulation.

EXPERIMENTAL PROGRAM

The development effort consisted of four distinct but interrelated phases:

Phase I: Evaluation of various ceramic fibers as replacements for fibrous potassium titanate in M-31 type insulation.- Ceramic fibers were

evaluated as replacement materials for fibrous potassium titanate (FPT) in M-31 insulation. The fibers which were evaluated included Fiberfrax, Refrasil, and Micro-Quartz. Selected properties of these fibers are compared to FPT below.

Property	Fiberfrax (washed bulk form)	Refrasil (SS1647)	Micro-Quartz (# 108)	FPT (block form)
Chemical composition (approx)	$3Al_2O_3 \cdot 2SiO_2$	SiO_2	SiO_2	$K_2Ti_6O_{13}$
Melting point (°F)	3200	3000	3000	2500
Fiber diameter (microns)	2	1	1.2	1

Candidate compositions were prepared from each of the above ceramic fibers by blending with asbestos fibers (90 percent ceramic fibers - 10 percent asbestos fibers) and combining the resulting mixtures with Ludox HS to form trowelable compositions. (Since this series of compositions was prepared to evaluate other fibers as replacements for FPT, the sample batches were identified as R-formulations. The exact formulations of these compositions and all other candidates evaluated in this program are shown in Table I.) The resulting matrices were applied to selected metal substrates and evaluated with respect to wet workability, drying characteristics, and insulating efficiency. (The insulating efficiencies of these insulations and all other compositions evaluated during this study are illustrated in Table II.)

The Refrasil composition (R-1) demonstrated good wet workability and satisfactory drying characteristics. Also, its insulating efficiency was satisfactory, but the coating shrank severely during testing, resulting in catastrophic failure of the coating in the form of cracking and separation from the substrate.

The Micro-Quartz (R-2) compositon was very difficult to work, i.e., it was extremely difficult to wet the fibers with Ludox HS colloidal silica. The drying characteristics of the coating appeared to be satisfactory; however, its insulating efficiency was not satisfactory. The coating exhibited an objectionable degree of shrinkage during radiant heat-testing, causing drastic deformation of the metal substrate. Therefore, Micro-Quartz was disqualified as a substitute for FPT.

Two forms of Fiberfrax fibers were evaluated as replacements for FPT, a washed bulk form and a chopped bulk form. The two forms of fibers are identical except that unfiberized glass particles have been removed from the washed bulk form. Both Fiberfrax compositions

exhibited good wet workability and satisfactory drying characteristics. The composition containing the washed bulk form of fibers (R-3) demonstrated superior insulating characteristics in comparison to the composition containing the chopped bulk form of fibers (R-3C). Although the insulating efficiency of the R-3 insulation was inferior to M-31, it was sufficient to qualify the washed bulk form of Fiberfrax fibers as a partial replacement for FPT.

Phase II: Evaluation of various colloidal silicas as binders for M-31 type insulations. - In an attempt to improve the texture and mechanical strength of M-31 without adversely affecting its insulating efficiency, other colloidal silicas were evaluated as substitutes for Ludox HS. Colloidal silicas which were evaluated include Nalcoag 1034A, 1050, and 1060, and Ludox HS 40. The properties of these colloidal silicas are compared with the properties of Ludox HS in the following table:

Property	Ludox HS	Ludox HS 40	Nalcoag 1034A	Nalcoag 1050	Nalcoag 1060
Percent silica	30	40	34	49	50
pH at 25°C	9.8	9.7	3.1	9.0	8.5
Particle diameter (m/?)	12	12	16-22	17-25	40-60
Surface area (M^2/g)	220-235	220-235	139-190	120-176	50-75
Specific gravity (25°C)	1.211	1.303	1.23	1.385	1.39
Viscosity (cps at 25°C)	5	34	5	70	5-10
Percent Na_2O	0.32	0.42	0.01	0.30	0.10

Ludox HS 40 was selected as a candidate binder to provide a sol with a higher silica content than Ludox HS without an increase in particle size; whereas, Nalcoags 1050 and 1060 provide sols with an increase in both silica content and particle size. Nalcoag 1034A was used to study the behavior of an acid silica sol for use in binder applications. (For identification purposes, this series of compositions evaluating binders was designated as B-formulations, Table I.)

Candidate compositions for evaluation were prepared from the same filler materials and by the same procedures used for M-31, except for the substitution of other silica binders for Ludox HS. Since the

wetting characteristics of the colloidal silica sols differ, it was necessary to use different amounts of each binder to obtain compositions with good wet workability. Upon preparation of the candidate insulations, they were applied to metal substrates and evaluated for texture and insulating efficiency. The texture of all of the experimental insulations was similar to that of M-31 in that they had a hard, strong exterior and a soft, weak interior caused by silica migration. The insulating efficiency of the coating bonded with Nalcoag 1034A was acceptable. However, this product had a pH of only 3.1 which caused severe corrosion of the metal substrate, therefore, it was not considered acceptable on this basis. The insulating efficiency of the coatings prepared with Nalcoags 1050 and 1060 was inferior to that of M-31, and did not warrant consideration as substitutes for Ludox HS. The bulk density of the insulation prepared with Ludox HS 40 was approximately 15 percent greater than that of M-31; therefore, its use as a heat shield insulation would impose a weight penalty. Based upon the above results, none of the products surveyed offered any competitive advantage over Ludox HS which was consequently used as the binder in all other test formulations.

Phase III: Gelation of Ludox HS as binder for M-31.- Since the texture of M-31 could not be improved by substituting other colloidal silica binders for Ludox HS, a study was made to evaluate methods of gelling the Ludox HS to retard silica migration, and thereby strengthen the insulation's interior region where substrate attachment occurs. The method of gelling Ludox HS which effectively retarded silica migration is described by Seitzinger (ref. 2). This gelling technique was used to develop an insulation (M-31X) having texture and adherence characteristics superior to M-31. However, to obtain an effective gel by this technique, the pH of the Ludox HS must be lowered to 2.5-3.0. Insulations prepared with this lower pH Ludox severely corroded some substrates, and the insulation became discolored by the corrosion products. Also, these insulations, in the wet condition, are stiff and difficult to apply.

For the above reasons, a new technique was selected for gelling the Ludox HS sol. The technique consists of acidifying the sol to a pH of 6.0. At this pH level, as shown in FIG 1, the sol gels in approximately 3-1/2 hours. The acidified sol was used to prepare an insulation similar to M-31. The composition of this insulation, designated as M-31G, is shown in Table I. The procedure used for curing M-31G after application was as follows:

a. Cover the wet material with an impervious plastic sheet for 24 hours.

b. Remove the plastic sheet and air-dry for 48 hours.

c. Raise the temperature of the insulation's substrate from room temperature to 49°C (120°F) in a 1-hour period, and hold at this temperature for 8 hours.

d. Raise the temperature to 83°C (180°F) over a period of 4 hours and hold at this temperature for 2 hours.

By covering the wet material for 24 hours sufficient time was allowed for complete gelation of the sol. Air drying removes the water slowly, thus minimizing cracking of the coating during drying. Finally, heat curing insures complete removal of the water. The texture of the M-31G was essentially uniform, indicating very little silica migration. The insulating efficiency of M-31G was comparable to M-31 as shown in Table II.

A flatwise tensile test was used to compare the adherence of M-31, M-31X, and M-31G as applied to stainless steel honeycomb sandwich substrates. The honeycomb substrates used for these tests consisted of a 1-inch thick honeycomb core, with 1/4-inch cell openings, and were brazed between two 0.010-inch thick face sheets. A 1/4-inch high open-faced honeycomb reinforcement, with 1/2-inch cell openings, was brazed to one face sheet of the basic honeycomb structure. The reinforcement, crushed to selected heights, was used for attaching the insulations. Approximately 0.3-inch thick coatings of the insulations were applied to the open-faced honeycomb substrates. The results of the flatwise tensile tests are illustrated in Table III, which show that gelling the silica sol is an effective method of improving the adherence characteristics of M-31 type insulations.

The drying characteristics of M-31G prepared from one batch of FPT were very satisfactory, as determined by coating a 15-inch square honeycomb panel. Virtually no surface cracks were present in the cured material. However, when another batch of FPT was used, large cracks developed in the coatings during drying. These cracks resulted from variations in the physical properties of the FPT caused by the manufacturer's in-process reduction of fiber size. Efforts to prevent the cracking were unsuccessful. Consequently, it was recognized that the composition of the insulation would have to be altered to increase its resistance to cracking.

Phase IV: Evaluation of insulations composed of Fiberfrax, fibrous potassium titanate, and asbestos bonded with gelled Ludox HS.- Since variations in the physical properties of FPT compromised an otherwise promising insulation, a study was made to evaluate Fiberfrax fibers as a partial replacement for FPT. This decision was based upon results obtained in Phase I of this program. Compositions were prepared by varying the proportions of Fiberfrax, FPT, and asbestos ingredients.

(This series of compositions was identified as FTA-(serial No.) formulations, where F, T, and A represent Fiberfrax, potassium titanate, and asbestos, respectively, and the three-digit serial number represents the proportions of F, T, and A ingredients, respectively.) All formulations were gelled by using the same gelling techniques that were used for M-31G. The resulting insulations were evaluated with respect to wet workability, drying characteristics, and insulating efficiency. Based upon these results, two insulations, FTA-983G and FTA-442G, were selected for further evaluation. The adherence of these insulations, as applied to open-faced honeycomb substrates, was determined by a flatwise tensile test. As shown in Table III, the flatwise tensile strength of the FTA-442G insulation is slightly superior to FTA-983G.

Although the gelling technique which was used to prepare the above insulations was effective in retarding silica migration, and in improving the flatwise tensile strength and related substrate adhesion, it was recognized that in practical applications, a gel time of 3-1/2 hours would not allow sufficient time to apply the insulation. A gel time of 5-1/2 hours was selected as a more realistic goal. This was accomplished by adjusting the pH of the Ludox HS to 6.6. The effect of extending the gel time on the adherence of the FTA-983G and FTA-442G insulations was determined. (Although the extension of gel time of the FTA-983G and FTA-442G insulations did not change their compositions, they were recoded as FTA-983 and FTA-442 when prepared with Ludox HS having a pH of 6.6.) The results of the flatwise tensile strength determinations, Table III, show that extending the gel time had a slight adverse effect on the adherence of both insulations. These results indicate that rapid gelation favors higher flatwise tensile strengths.

In an attempt to improve the flatwise tensile strength of the FTA-442 insulation, finely divided asbestos fibers were added to the Ludox HS binder to serve as a gel reinforcement. The fine-particle asbestos had an opposite effect which resulted in severe cracking of the insulation (FTA-442R). A possible explanation of this adverse effect is that the gel was reinforced to the extent that it would not yield readily to stresses imposed during shrinkage as drying occurred, and the coating cracked to relieve the stresses. A further disadvantage was evident in that the insulating efficiency of FTA-442R was inferior to FTA-442.

Based upon the results obtained in the experimental phases of this program, FTA-442 insulation was considered a qualified substitute for M-31. However, subsequent to the successful development of FTA-442 insulation, the manufacturer advised that FPT would be removed from the commercial market, and that they had developed a new material,

pigmentary potassium titanate (PKT), as a replacement for FPT. The chemical composition of the two materials is essentially identical - approximately $K_2Ti_6O_{13}$. Efforts to substitute PKT for FPT in the FTA-442 formulation resulted in the development of a suitable alternate material for M-31.

The new material has been designated as FTA-442A insulation. Another material, designated FTA-532A insulation, was developed concurrently with FTA-442A and is also considered a suitable replacement for M-31.

RESULTS AND DISCUSSION

Engineering Properties

To evaluate the capabilities of the FTA insulations, it was necessary to determine several of their properties. Properties of engineering interest that were determined are as follows:

a. Bulk density and water absorption

b. Density gradient

c. Mechanical strength

d. Refractoriness

e. Specific heat

f. Thermal conductivity

g. Thermal-shock resistance

h. Reflectance

i. Moisture resistance.

Unless otherwise noted, all property determinations were made on the unreinforced material, i.e., not applied to a metal substrate.

Bulk density and water absorption.- The bulk density and water absorption were determined according to ASTM Designation C20-46, "Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick," on FTA materials approximately 3/8-inch thick. The bulk density and water absorption of the

FTA insulations are compared to M-31 in the following table.

	Bulk Density (lbs/ft^3)	Water Absorption (percent)
M-31	55	69
FTA-442A	61	44
FTA-532A	59	45

Density gradient.- The density gradients through 0.35-inch thick specimens of the FTA insulations are shown in FIG 3, and are compared to M-31. The density gradients were determined by grinding off thin layers (approximately 0.020-inch) of the specimens and calculating the density of each layer removed. It is evident, from FIG 3, that the technique used to gel the Ludox HS binder in the FTA insulations was effective in retarding silica migration, which is the prime cause of density gradients in materials of this type.

Mechanical strength.- The mechanical strengths were determined by means of a transverse (flexure) test. The measurements were made on materials approximately 1/3-inch thick. The strengths of the FTA insulations under various conditions are compared to M-31 below.

	Modulus of Rupture (psi)		
Conditions	M-31	FTA-442A	FTA-532A
Load applied face side	670(101)	1535(292)	1290(139)
Load applied back side	1172(124)	1380(225)	1245(139)
Soaked in water 100 hours, load applied back side	926(190)	965(114)	875(117)
Soaked in water 100 hours, dried, load applied back side	1159(118)	1340(144)	1235(148)
Heated at 24 Btu/ft^2-sec for 150 seconds, load applied back side	995(120)	920(83)	847(70)

Each modulus of rupture value is an average calculated from 15 specimens. The numbers in parentheses are average deviations from the average modulus of rupture values. The large deviations are caused by the presence of voids in the materials, an inherent characteristic of these materials. The results show a moderate difference in the strength of the exterior and interior of the FTA insulations, the

interior being the strongest. This indicates that there is some silica migration toward the substrate-insulation interface during gelling of the silica sol and/or drying of the insulation. This indication is substantiated by the density gradient determinations (FIG 3) which show that the interior of the insulations is slightly denser than the exterior. (Note the extreme opposite effect for M-31, which accounts for its weak interior.) By soaking the FTA insulations in water for 100 hours their strength is reduced by approximately 30 percent, as compared to 20 percent for M-31. However, their strength can be readily restored by drying the insulations to remove the absorbed water. The results show that the FTA insulations lose approximately 32 percent of their strength, as compared to 15 percent for M-31, when exposed to a radiant heat flux of 24 Btu/ft^2-sec for 2-1/2 minutes. The reduction in strength is primarily attributed to the effect of heat on the asbestos and Fiberfrax fibers. These fibers become brittle and lose much of their strength when exposed to elevated temperatures. In fact, it is known that Chrysotile asbestos fibers, of the grade used in the FTA and M-31 insulations, lose approximately 27 and 65 percent of their strength when heated for three minutes at 317°C (600°F) and 650°C (1200°F), respectively. The surface temperature of the FTA and M-31 insulations is approximately 760°C (1400°F) after 2-1/2 minutes exposure to a radiant heat flux of 24 Btu/ft^2-sec. Since the FTA insulations contain more asbestos fibers than M-31, and since they also contain Fiberfrax fibers, they would be expected to lose more of their strength when exposed to elevated temperatures.

Refractoriness.- An indication of the refractoriness of the FTA insulations was obtained by heating rectangular specimens (1" x 6" x 0.3") in an electric furnace at a rate of 111°C (200°F) per hour. The bars were cantilevered to allow bending when softening occurred. For comparison, M-31 specimens were included in the test. At 871°C (1600°F), all specimens had softened sufficiently to cause slight bending. Starting at this temperature, specimens were removed from the furnace at 56°C (100°F) temperature intervals and examined visually. At 1204°C (2200°F), the interior of the M-31 showed some melting. At 1315°C (2400°F), there was considerable melting of the M-31, while the FTA insulations showed only slight melting. The test was terminated at this temperature.

Specific heat.- Specific heat was determined by the method of mixtures using a water calorimeter. In the 25-100°C (77-212°F) temperature range, the specific heat of the FTA insulations was determined to be 0.246 Btu/lb-°F. In the same temperature range, the specific heat of M-31 is 0.31 Btu/lb-°F.

Thermal conductivity.- Thermal conductivity was measured with a guarded hot plate. The thermal conductivities of the FTA insulations

are compared to M-31 in the following table.

Temperature °C	Temperature °F	K(Btu/ft^2/hr/°F/in) FTA-442A	FTA-532A	M-31
93	200	1.34	1.38	0.95
204	400	1.52	1.68	1.11
315	600	1.67	1.77	1.23
426	800	1.77	1.80	1.30

Thermal-shock resistance.- Thermal-shock resistance was determined on both reinforced and unreinforced specimens of FTA insulations. The reinforced specimens were prepared by applying 0.3-inch thicknesses of the materials to stainless steel substrates (0.04" x 4" x 6") overlaid with stainless steel expanded metal. The unreinforced specimens were 0.3-inch thick, 4 inches wide, and 6 inches long. Three specimens each of the reinforced and unreinforced materials were tested. Each specimen was exposed to a radiant heat flux of 24 Btu/ft^2-sec for three minutes and quenched immediately in water. There was no evidence of thermal-shock failure of any specimen tested, indicating that the thermal-shock resistance of the FTA insulations is comparable to that of M-31.

Reflectance.- A Perkin-Elmer Model 112 double-pass, single-beam spectrophotometer equipped with a special integrating sphere was used to measure absolute spectral reflectance in the 0.30-2.2 micron wavelength range. The reflectances of the FTA insulations are illustrated and compared to M-31 in FIG 4.

Moisture resistance.- The moisture resistance of the FTA insulations was determined by a boiling water test. Specimens of the insulations were immersed for three hours in boiling tap water which had a pH of 7.3. There was no appreciable coloration of the water during the test which indicated that water had little effect on the stability of the materials.

Insulating Capabilities

Radiant heat.- In addition to preliminary results shown in Table II, the insulating capabilities of the FTA insulations were further evaluated as applied to the S-IC heat shield substrate. FTA insulations (0.3-inch thick) were applied to panels of the heat shield substrate

and exposed to radiant heat fluxes of 24 and 40 Btu/ft^2-sec for 180 seconds. Chromel-alumel thermocouples were attached to the back side of each specimen for monitoring temperature rise. The insulating qualities of the FTA insulations are illustrated and compared to M-31 in FIG 5 and 6. These data show that the FTA insulations are better insulators in radiant heating environments than M-31, especially at the higher heat flux of 40 Btu/ft^2-sec. The superior insulating qualities of the FTA insulations, in radiant heating environments, are primarily attributed to their superior reflectance at elevated temperatures. Throughout the entire test program, it was noted that insulations composed primarily of potassium titanate turned yellow during radiant heat testing. The degree of color change was directly related to the potassium titanate content; the FTA insulations exhibited a smaller color change than M-31. Consequently, the superior reflectance stability of the FTA insulation makes them better insulators in radiant heating environments than M-31. Based upon this reasoning, it would appear that FTA-532A should be a better insulator than FTA-442A in radiant heating environments. However, this is not the case, FTA-532A does not contain sufficient PKT to provide the requisite infrared reflectance.

Since FTA-442A proved to be the superior insulator, its insulating capabilities in radiant heat were determined at heat fluxes beyond the maximum capability of M-31, which is about 70 Btu/ft^2-sec. For this determination, FTA-442A insulation, 1/2-inch thick, was applied to mild steel substrates (0.06" x 6-1/2" x 6-1/2") overlaid with expanded metal. A chromel-alumel thermocouple was attached to the back side of each specimen to monitor temperature rise. The specimens were exposed to a radiant heat flux of 80 Btu/ft^2-sec for 180 seconds. At the end of the tests, the average back face temperature rise for FTA-442A insulation was 292°C (525°F) as compared to 416°C (750°F) for M-31. Since 80 Btu/ft^2-sec is close to the upper limit of the test equipment (ref. 3) for long duration testing, the insulation was not tested at higher heat fluxes.

Convective heat.- The insulating capabilities of FTA-442A insulation were further evaluated in convective heating environments. An oxygen-acetylene-butane-air blast burner was used as the heat source. The test specimens consisted of varied thicknesses of insulation applied to mild steel blanks (0.04" x 4" x 6") overlaid with expanded metal. Chromel-alumel thermocouples were attached to the back side of each specimen for monitoring temperature rise. The insulating capabilities of FTA-442A are illustrated and compared to M-31 below.

Heat Flux (Btu/ft^2-sec)	Insulation Thickness (in.)	Back Face Temperature Rise (°F) 145 Seconds M-31	145 Seconds FTA-442A	180 Seconds M-31	180 Seconds FTA-442A
10	0.310	301	315	372	411
30	0.310	579	589	690	731
50	0.450	609	298	756	439
90	0.450	959	466	1124	696

The results show that the insulating efficiency of FTA-442A insulation in convective heating environments is comparable to M-31 at the lower heating rates (10 and 30 Btu/ft^2-sec). However, at the higher heating rates, FTA-442A insulation is considerably superior to M-31. This is attributed to the fact that the FTA-442A insulation is more refractory than M-31, therefore, it is capable of withstanding higher temperatures.

CONCLUSIONS

A new material has been developed as an alternate and/or replacement for M-31, the material presently used for insulating the base heat shields of the S-IB and S-IC stages of the Saturn vehicles. The material, designated as FTA-442A insulation, is a low density, highly reflective, unfired, ceramic insulating coating comprised of alumina-silica fibers, pigmentary potassium titanate, and asbestos fibers bonded with a colloidal silica sol. The silica sol is gelled to retard silica migration during drying of the coating. FTA-442A insulation has the following properties:

a. Bulk density - 61 lbs/ft^3

b. Water absorption - 44 percent

c. Modulus of rupture - 1535 psi

d. Absolute spectral reflectance - 83.5 percent at 1.7 microns

e. Specific heat - 0.246 Btu/lb-°F in the 25-100°C (77-212°F) temperature range

f. Thermal conductivity - 1.34 and 1.77 Btu/ft^2/hr/°F/in at 93°C (200°F) and 426°C (800°F), respectively.

g. Melting point - approximately 1315°C (2400°F), and good thermal-shock resistance.

The insulating qualities of FTA-442A insulation, in both radiant and convective heating environments, are superior to M-31. A 0.3-inch thick coating of the insulation applied to a panel of the S-IC type heat shield substrate and exposed to a radiant heat flux of 24 Btu/ft^2-sec for 145 seconds had a back-face temperature rise of only 64°C (115°F) as compared to 105°C (189°F) for M-31. When exposed to a radiant heat flux of 40 Btu/ft^2-sec for 145 seconds, the back-face temperature rise was 224°C (404°F) for the FTA-442A and 274°C (494°F) for M-31. FTA-442A insulation is capable of insulating against radiant heat loads of at least 80 Btu/ft^2-sec for three minutes, whereas, M-31 degrades rapidly under the same conditions. FTA-442A insulation is also an effective insulator against convective heating environments. A 0.31-inch thick coating of FTA-442A applied to a mild steel substrate, 0.04-inch thick, and exposed to a convective heat flux of 10 Btu/ft^2-sec for 145 seconds, had a back face temperature rise of 175°C (315°F) as compared to 167°C (301°F) for M-31. A 0.45-inch thick coat of FTA-442A, applied to a mild steel substrate, 0.04-inch thick, and exposed to a convective heat flux of 90 Btu/ft^2-sec for 145 seconds, had a back face temperature rise of 259°C (466°F). This compares to a temperature rise of 533°C (959°F) for M-31.

Gelling the silica sol to retard silica migration during drying of the FTA-442A insulation produces a material of mechanical strength and related substrate adhesion which is considerably superior to M-31. The adhesion of FTA-442A applied to the S-IC type heat shield substrate is approximately four times that of M-31 as measured by a flatwise tensile test.

Based upon the results of this program, both FTA-442A and FTA 532A insulations are suitable replacements for M-31. FTA-442A is superior to FTA-532A with respect to insulating capabilities and adherence characteristics and is recommended as the replacement material for M-31.

TABLE I.- COMPOSITIONS OF CANDIDATE INSULATIONS

Composition	Formulation																		
Fillers	R-1	R-2	R-3	R-3C	B-1	B-2	B-3	B-4	M-31	M-31X	M-31C	FTA-541C	FTA-983C	FTA-442C	FTA-983	FTA-442	FTA-442R	FTA-532A	FTA-442A
Fibrous Potassium Titanate					90	90	90	90	90	90	90	40	40	40	40	40	40		
Asbestos Fibers	10	10	10	10	10	10	10	10	10	10	10	10	15	20	15	20	20	20	20
Refrasil (SS 1647)	90																		
Micro-Quartz (No. 108)		90																	
Fibertrax (Washed Bulk)			90									50	45	40	45	40	40	50	40
Fiberfrax (Chopped)				90															
Pigmentary Potassium Titanate																		50	40
Binders*																			
Ludox HS	850	610	190	265					350										
Ludox HS (pH 6.0)											350	350	350	350					
Ludox HS (pH 6.6)															350	350	350**	350	350
Ludox HS (2.3)										350									
Nalcoag 1034A								350											
Nalcoag 1050						270													
Nalcoag 1060							225												
Ludox HS 40					350														

NOTES: * Amount of binder is given as bulk volume (cubic centimeters) per 100 parts (grams) of filler materials.

**Contains 1/2 percent (dry weight basis) of fine particle asbestos fibers.

TABLE II.- INSULATING EFFICIENCY OF CANDIDATE INSULATIONS

Compositions	Insulation Thickness (in.)	Back-Face Temperature Rise (°F) 145 Seconds	180 Seconds
R-1	0.365	260	365
R-2	0.310	448	540
R-3	0.310	302	443
R-3C	0.250	620	763
B-1	0.290	281	346
B-2	0.300	326	428
B-3	0.330	327	425
B-4	0.240	318	408
M-31	0.290	270	327
M-31X	0.260	311	389
M-31G	0.300	273	361
FTA-541G	0.300	273	370
FTA-983G	0.280	278	362
FTA-442G	0.300	282	368
FTA-983	0.280	246	305
FTA-442	0.300	257	320
FTA-442R	0.290	334	420
FTA-532A	0.300	311	387
FTA-442A	0.300	237	323

NOTES: The substrates for the insulations were stainless steel blanks (0.040" x 6" x 11") overlaid with stainless steel expanded metal.

$Q = 24$ Btu/ft^2-sec (radiant).

TABLE III.- FLATWISE TENSILE STRENGTH OF CANDIDATE INSULATIONS

Compositions	Reinforcement Crimp Height (in)	Flatwise Tensile Strength (psi)
M-31	0.145	12.6
M-31	0.188	36.7
M-31X	0.145	43.5
M-31X	0.188	55.4
M-31G	0.145	53.5
M-31G	0.188	63.7
FTA-442G	0.113	41.3
FTA-983G	0.113	35.2
FTA-442	0.113	35.2
FTA-983	0.113	26.0
FTA-442	0.145	45.7
FTA-442	0.188	60.6
FTA-532A	0.113	40.0
FTA-532A	0.145	48.0
FTA-442A	0.113	45.0
FTA-442A	0.145	60.9

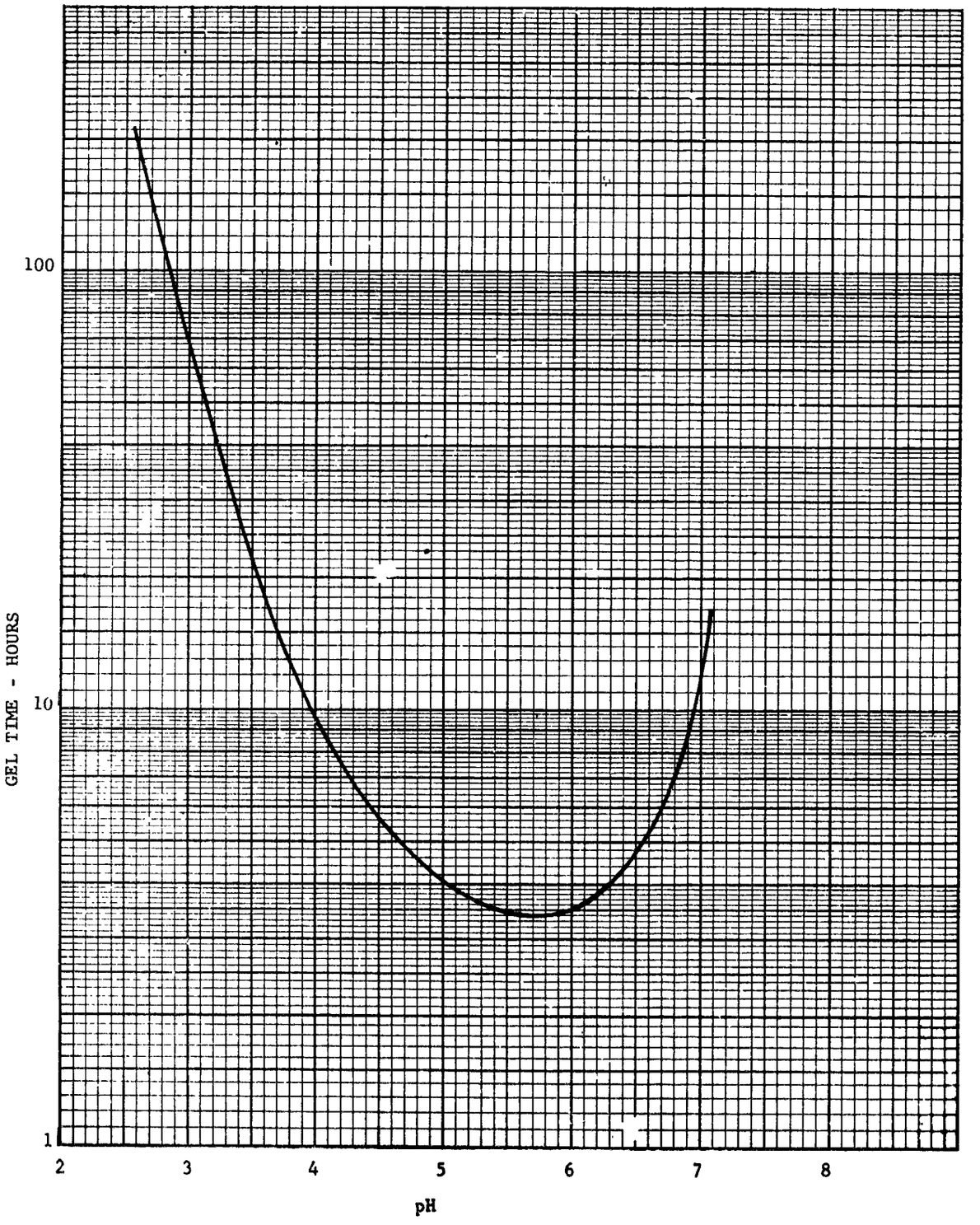

FIGURE 1.- GELATION OF LUDOX HS AT ROOM TEMPERATURE

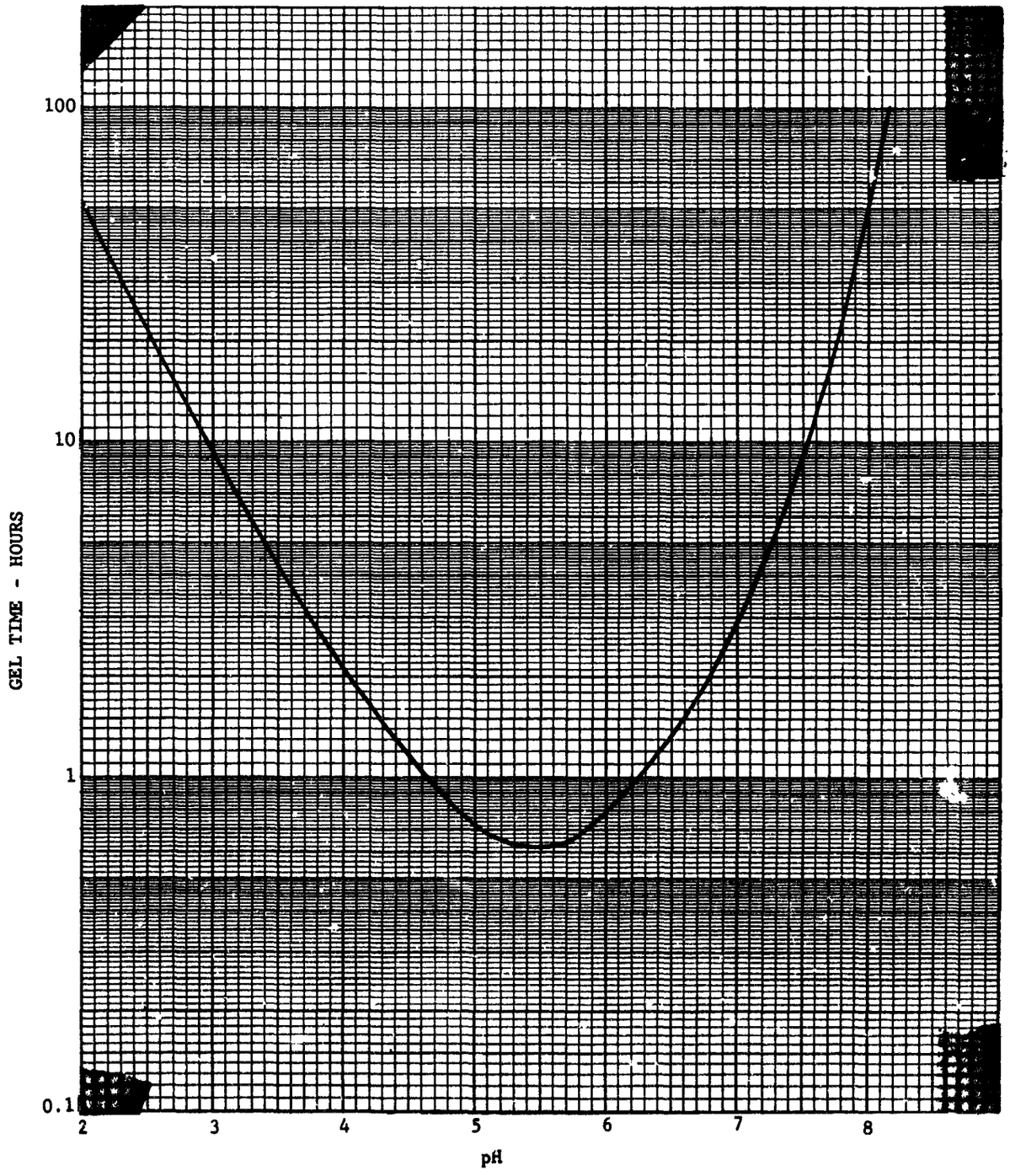

FIGURE 2.- GELATION OF LUDOX HS AT 140°F

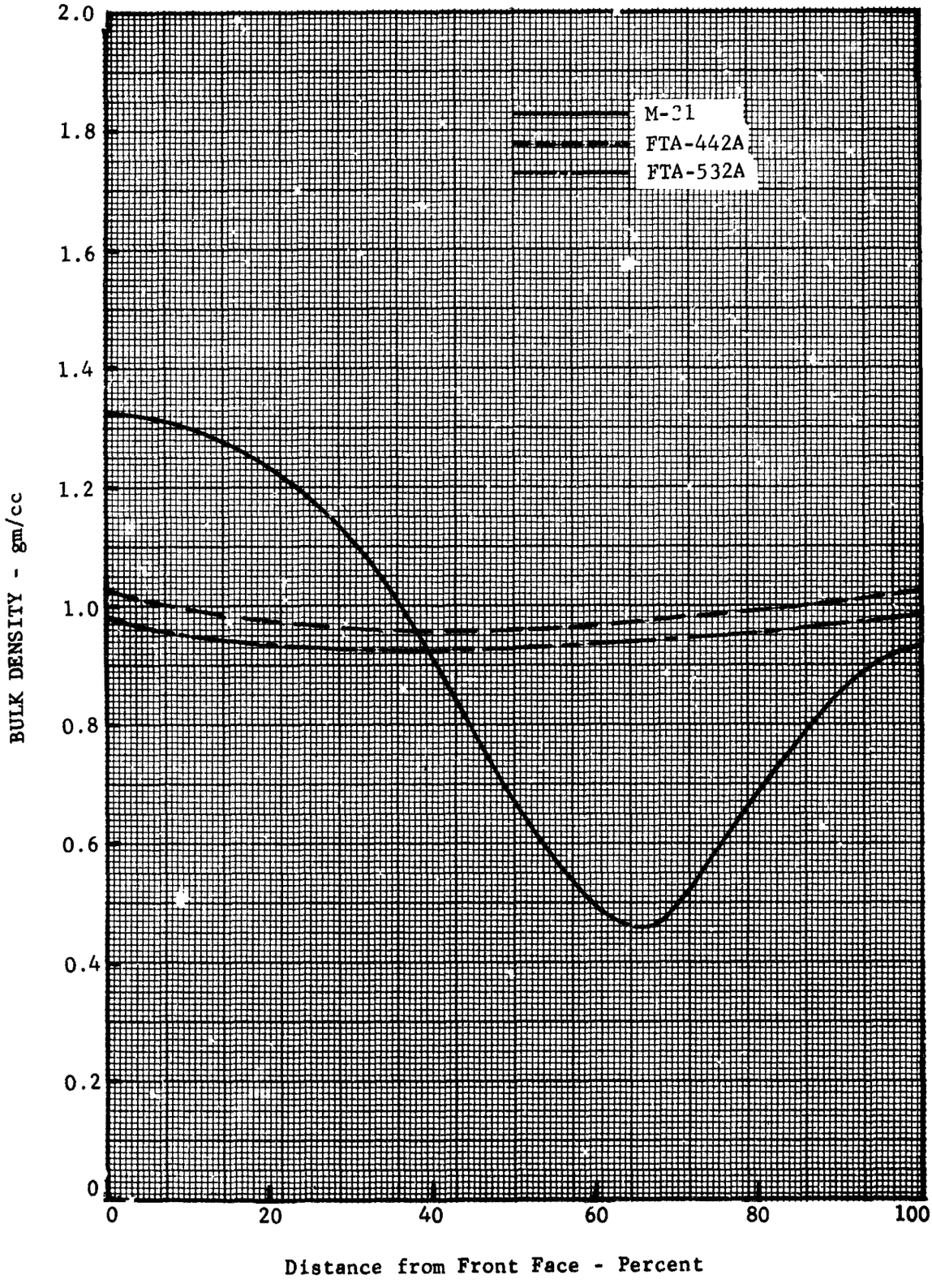

FIGURE 3.- COMPARISON OF DENSITY GRADIENT THROUGH M-31 AND FTA INSULATION

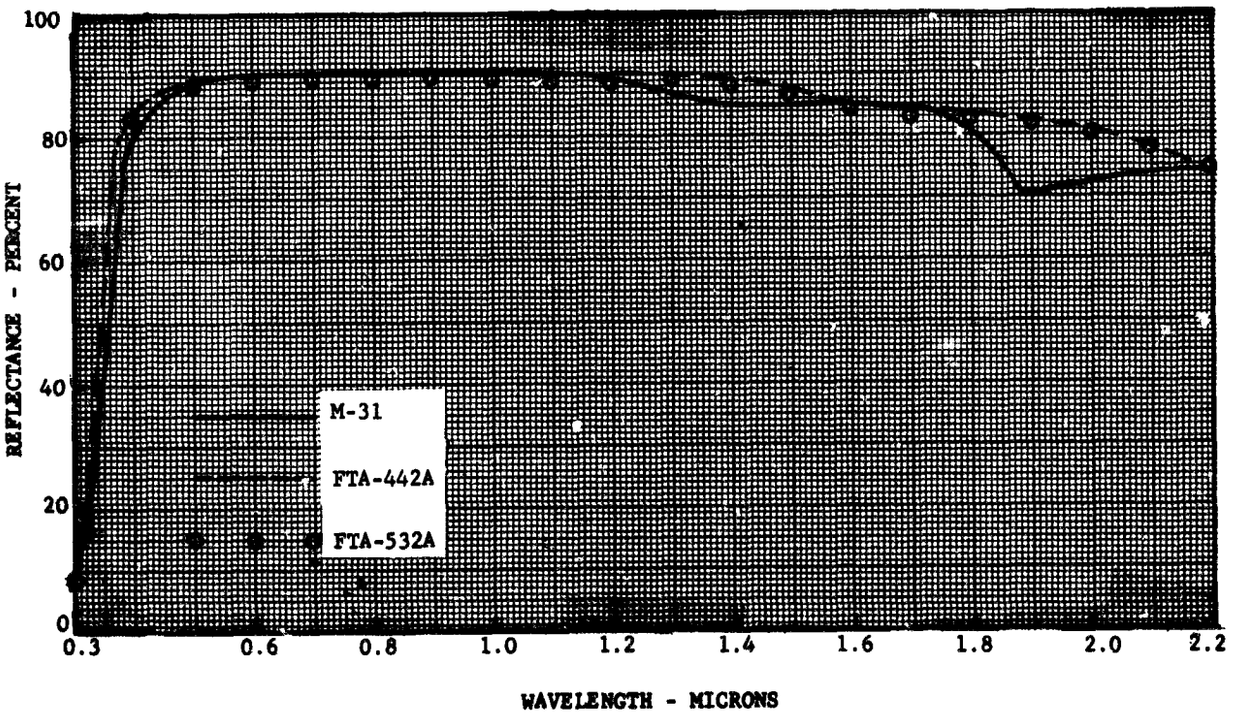

FIGURE 4.- ABSOLUTE SPECTRAL REFLECTANCE OF FTA AND M-31 INSULATIONS

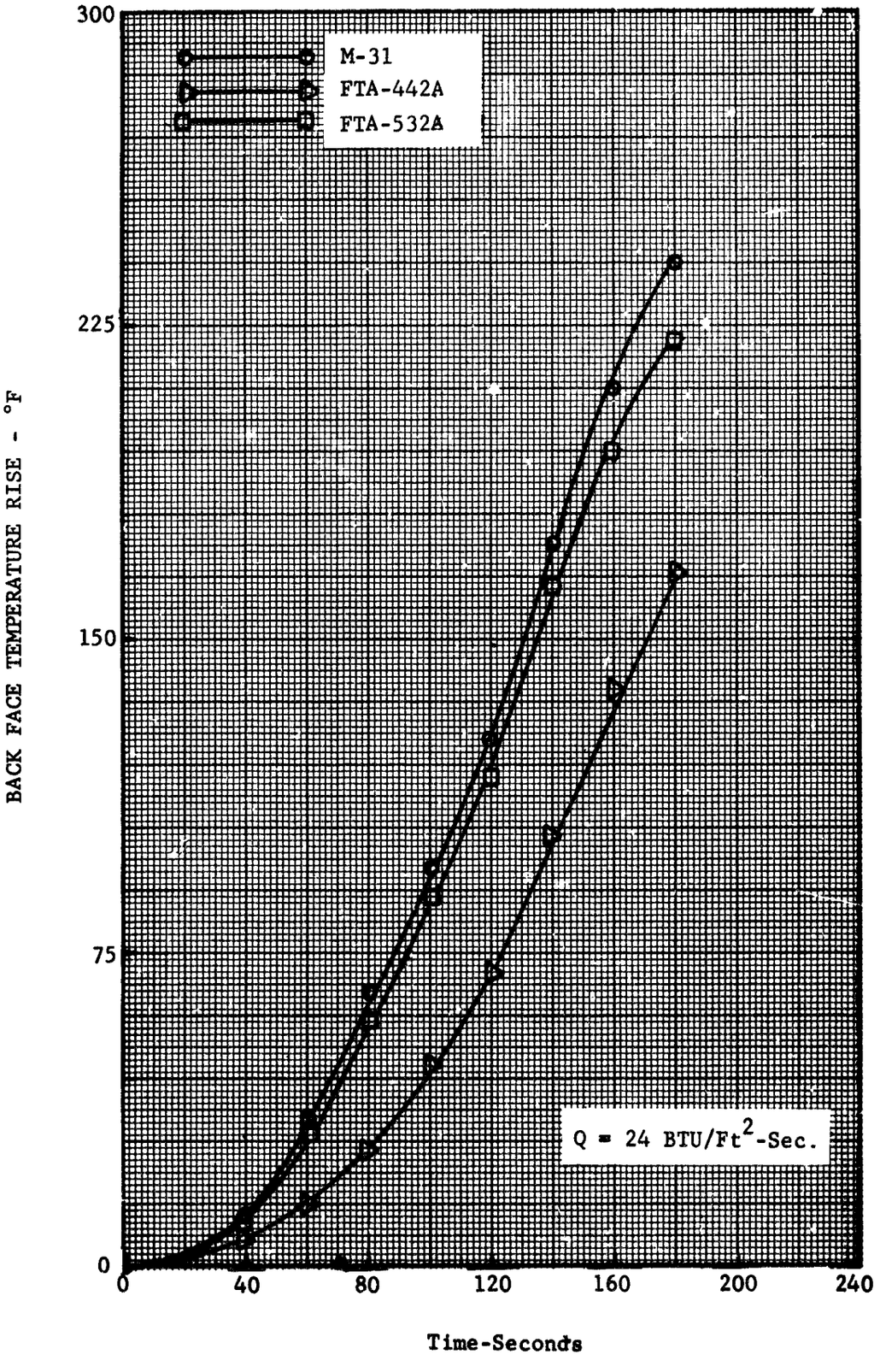

FIGURE 5.- COMPARISON OF INSULATING EFFICIENCY OF M-31 AND FTA INSULATIONS EXPOSED TO RADIANT HEAT FLUX OF 24 Btu/ft^2-Sec.

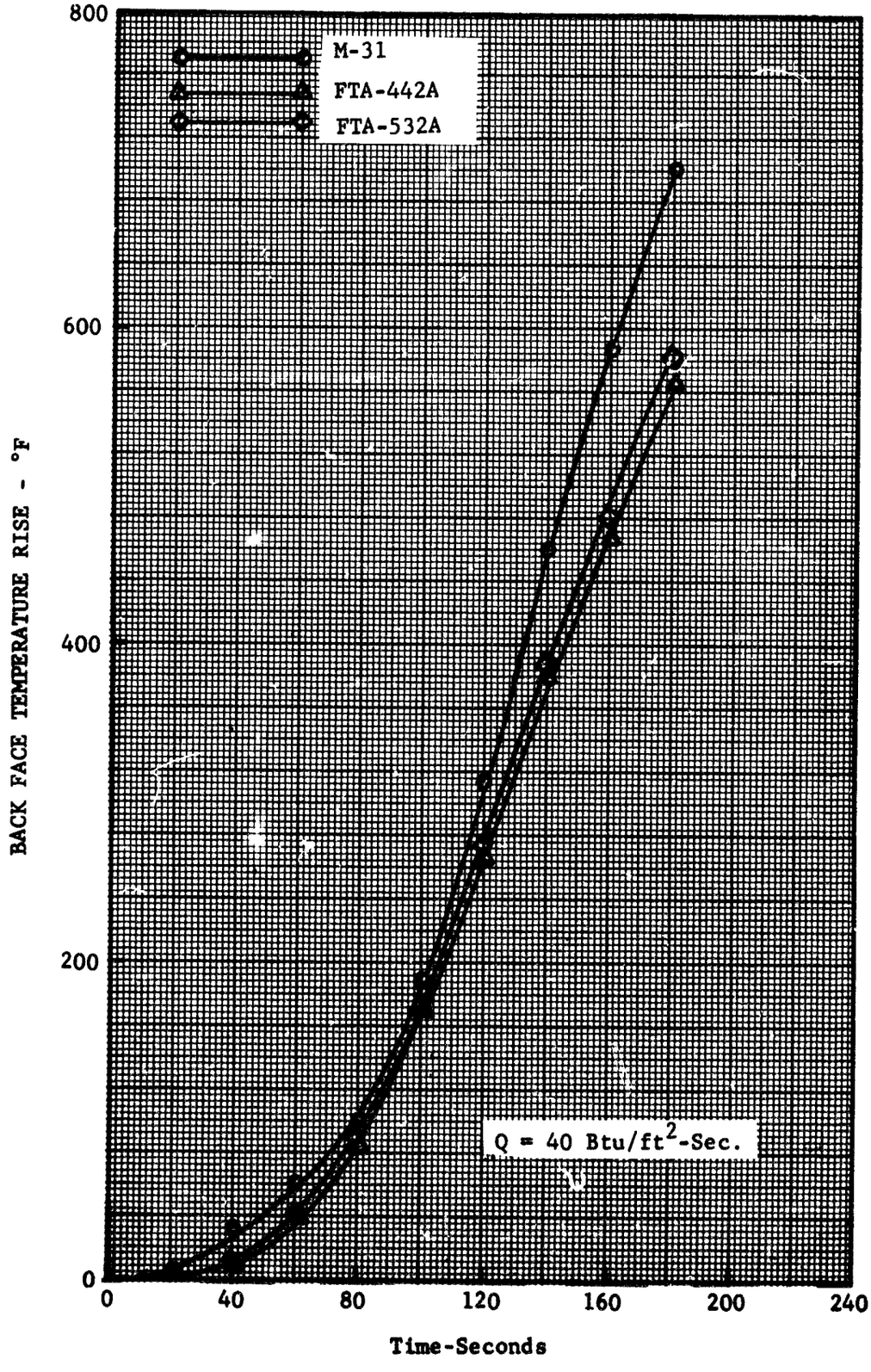

FIGURE 6.- COMPARISON OF INSULATING EFFICIENCY OF M-31 AND FTA INSULATIONS EXPOSED TO RADIANT HEAT FLUX OF 40 Btu/ft^2-Sec.

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THERMAL INSULATIONS FOR LAUNCH VEHICLE RADIANT HEATING ENVIRONMENTS

By Vaughn F. Seitzinger

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