DYNATECH CORPORATION

THE THERMAL CONDUCTIVITY OF TWO THERMAL INSULATING MATERIALS

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Progress through Research

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This report covers work carried out for the National Aeronautics and Space Administration, George C. Marshall Space Flight Center, Huntsville, Alabama, under Contract Number NAS 8-21262.

I - Objective of Investigation

To measure the thermal conductivity of two thermal insulating materials over the approximate temperature range -150°F to 200°F, in a vacuum using available equipment with a minimum of modification and to an accuracy of ±15% or better.

II - Materials and Desired Test Procedure

Two materials were supplied for evaluation:

(1) A low density polyurethane foam

(2) 0.25 mil aluminized Mylar with 30 mil polyurethane foam spacers. This multilayer cryogenic insulation was to be tested in thicknesses of 0.25 inches and 1.0 inches respectively.

The following desired test procedure was to be followed where possible: The test samples were to be assembled in the measuring system and conditioned at 250°F in helium for a period of one hour. They were to be evacuated to a pressure which should not exceed $8x10^{-6}$ torr during the test cycle. Thermal conductivity measurements were to be made at eight mean temperatures in the range -150°F to 200°F with a temperature differential not exceeding 15 degrees across the sample. After each successive determination the system was to be pressured to 760 torr with dry helium and then evacuated once again and the next measurement made. Once the sample had been installed it was not to be exposed to any atmosphere other than dry helium.

Reference: NAS - 23

III - Method Used

The method chosen was basically the guarded hot plate technique which is a standard technique (ASTM C 177-63) for the measurement of the thermal conductivity of thermal insulating materials with an accuracy of between ±2% and ±5%, depending upon the temperature and the conditions. The Dynatech TCFG Guarded Hot Plate Instrument series has been designed to conform to the specifications. Results obtained recently on the instrument used for this investigation during an International Round Robin sponsored by the International Institute of Refrigeration upon a sample of a fibrous insulation were within 2% of the accepted values as measured at two National Standards Laboratories over a major portion of the present temperature range.

This method, as devised, is normally used for measurement on homogeneous non-anisotropic thermal insulations such as the polyurethane foam. The cryogenic multi-layer insulations are greatly anisotropic in property since the conductivity parallel to the layers can be several orders of magnitude greater than that perpendicular to the layers. The whole performance of such an insulation depends upon the number of thermal resistances and radiation barriers restricting the heat flow perpendicular to the layers. Thus for the measurements on the multi-layer materials, the basic instrument and measurement techniques were modified considerably in order to obtain experimental conditions where good accuracy of measurement could be attained. Two other very important factors in relation to the thermal conductivity of the layer type insulation are the degree of vacuum in which the measurements are made and the degree of loading of the sample.

The guarded hot plate apparatus used consists of the following essential parts:
(1) A 4 inch central uniformly wire wound heater surrounded by a separate 2 inch wide annular uniformly wound heater with a 0.125 inch gap between them. The heater core is embedded into a stabilized silicone elastomer and sandwiched between two uniformly thick (0.5 inches) aluminum plates with blackened outer surfaces. At several radial positions small 0.015 inch square grooves are cut in the surfaces of the plates. Fine wire thermocouples of copper/constantan in multi-bore quartz protection tubing are

fixed rigidly into the grooves so that the temperature distribution over the whole plate can be evaluated.

An eight junction differential thermocouple is fixed between the core and annular ring of the heater. The output of this thermocouple is used to control the power applied to the annular guard heater.

(2) Two auxiliary heaters which are similar in shape and size to the main heater, except that each is one continuous heater and there is no annular air gap. The surfaces of these are blackened and also have grooves containing a number of thermocouples cut in the surfaces.

(3) Two heat sinks through which cooling fluid can be passed.

(4) An outer cylindrical tube approximately the length of the total sample stack which can be both cooled and heated to a controlled temperature.

The test sample consists of two 8 inch in diameter pieces of uniform thickness and good surface finish. More preferably these samples should be 4 inches in diameter surrounded by annular rings of slightly less than 2 inches wide such that a 0.125 inch air gap is obtained between them. In the present investigation the polyurethane was supplied in the latter form and the multi-layer insulation supplied as sheets in the same form from which test samples of the appropriate thickness could be fabricated in the apparatus.

Fine wire thermocouples were placed in very thin slots cut into the surface of each of the polyurethane foam samples such that the thermocouple beads were at the surfaces of the samples.

In operation, the main heater is sandwiched between the two samples. This combination is sandwiched between the two auxiliary heaters and heat sinks. The composite stack is mounted vertically and surrounded by the outer cylindrical tube. The whole assembly and surrounds is filled with dry thermal insulation powder of very low thermal conductivity and then covered with a vacuum jar.

Steady power from a highly stabilized dc supply is supplied to the core of the main heater. Stabilized power is supplied to the auxiliary heaters and cooling fluid, either water or liquid nitrogen, to the heat sinks and outer cylinder. Thus, the desired temperature distribution is set up in the system. The output of the differential thermocouple in the heater is fed to a dc amplifier which in turn activates an SCR power controller supplying stabilized power to the annular guard heater. In this way the temperature of the main heater is controlled so that the hot faces of each sample are constant, there is no radial heat loss, and the cold faces also are constant. At equilibrium conditions with the temperature differences across each sample controlled to be the same thickness, the power supplied to the main heater is assumed to be distributed equally between the two test discs.

At equilibrium conditions the power to the main heater is measured and the temperatues of the hot and cold faces of the sample obtained from thermocouples in the surfaces of the plates and sample. The thermal conductivity is derived from the power supplied, the temperature difference as measured and the known dimensions of the sample, the thickness being measured where possible in the apparatus prior to starting.

The tests on the polyurethane foam were carried out according to the above procedure combined with the imposed conditions regarding the atmospheric and heating conditions. Temperature differences of between 15 and 30 deg were used in the measurements.

For the multi-layer insulation the following modifications were used in order to reduce the overall effects of the anisotropy of the thermal conductivity upon the measurements:

(1) The samples were prepared and assembled very carefully in order to ensure that there was an 0.125 inch air gap between the 4 inch core and 2 inch annulus. Under these conditions, providing the center and annular sections were controlled at the same temperature ($\sim 0.1°$ or better) the problem of heat flow from the central section via radial conduction is greatly reduced.

(2) Additional thermocouples were fixed into the heater plates in order to monitor the temperatures more definitely.

(3) One of the normal auxiliary heaters was replaced with a main heater configuration very similar to the one described above. This was done to try to obtain really good temperatue control on both faces of one of the two individual test pieces.

(4) Very great time and care were taken to allow the apparatus to attain each steady temperature equilibrium. In some cases this was the order of three days after obtaining the requisite vacuum.

(5) The outer cylindrical heater was maintained by appropriate heating and cooling at a steady temperature equivalent to the mean temperature of the test sample.

(6) At each mean temperature tests were run with the temperatures of the guard and annular sections of the heater at the same mean value to better than 0.15 deg. It was found that by averaging all of the temperatures an exact zero difference could not be attained due to slight imperfections in the thermocouple wire. Tests were therefore attempted whereby a measurement was undertaken with this order of difference across the faces of the hot and cold plates. Without changing the other conditions of the test the "guard" temperatures on the hot face and one of the cold faces were adjusted manually so that the temperature difference across the faces was of the same order as before, but of opposite sign. This was done in order to obtain two "apparent" thermal conductivities at the same temperature for different heat loss conditions, one positive and the other negative. These "apparent" values could be plotted against the temperature difference and the value for zero difference taken as the thermal conductivity at that mean temperature.

Because of the criticallity that the loading of the sample has on the overall value of thermal conductivity which can be obtained, the following technique was used to avoid loading the sample with the weight of the stack. Since the bulk of the thickness of the sample consisted of the layers of foam, the outer annular edge of each sample was notched at four places in the circumference. Pieces of polyurethane foam were cut carefully to the same thickness as the test stack and placed in the notches to support the central and upper heater plates. The upper heat sink was supported separately so that it did not load the stack at all. While no absolute measure of the loading could be made, it was decided to adopt the method so that the expansion or contraction of the sample would be reproduced by the supports which were of the same material thus giving a reproducible load at each mean temperature.

IV - Results

The experimental results for the particular samples tested for the particular test conditions are given in Tables I and II, and are shown in Figures 1 and 2.

(a) Polyurethane Foam. The results were obtained in a vacuum of between 10^{-4} and 10^{-5} torr as indicated by a gauge in the system. A better vacuum could not be attained for this test, but since this material was of a "closed cell" type there was little likelihood that the overall thermal conductivity would be affected by a higher vacuum.

The results showed the characteristic inflection in the thermal conductivity versus temperature curve which is obtained for these materials. This is due to the liquification of the refrigerant gas contained in the pores as the temperature falls. The exact temperature at which it occurs and the magnitude of the change is dependent upon the refrigerant.

(b) Multi-layer Insulation. The results shown were obtained for an overall indicated vacuum of 10^{-5} torr or better except for the first and second experimental points on each sample when the pressure was still falling slightly in the range 5×10^{-4} to 10^{-5} torr.

The results for both thicknesses of sample may be said to be in good agreement considering that exact similarity in test conditions could not be attained in the case both of the gas pressure and applied pressure. The thermal conductivity of this type of material is so dependent upon these two factors that much larger differences could have been expected.

In each case it can be seen that the earlier experimental points of each series are much higher in value than the later ones. This is due solely to the effect of vacuum on the results since it was found that the system took a long time to attain a pressure of 10^{-5} torr or better.

TABLE I

Thermal Conductivity of a Polyurethane Foam In Vacuum

Measured Density : 1.8 lb ft^{-3}

Mean Temperature, °F	Thermal Conductivity, Btu hr ft^{-1} deg F^{-1}
151.9	0.0154
95.1	0.013
53.8	0.0118
31.5	0.0115
0.8	0.013
-36.3	0.0146
-95.2	0.013
-146.8	0.0115

Reference: NAS - 23

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TABLE II

Thermal Conductivity of Double Metallized Mylar-Foam Insulation in Vacuum

Sample: 6 layers of foam, 5 of Mylar

Mean Temperature, °F	10^4 Thermal Conductivity Btu hr ft^{-1} deg F^{-1}
130.9	10.0
154.1	2.9
98.2	1.7
48.6	0.9
-151.1	4.9
- 81.3	5.7
- 2.2	5.7
103.6	8.4
146.6	1.5

Sample: 24 layers of foam, 23 of Mylar

Mean Temperature, °F	10^4 Thermal Conductivity Btu hr ft^{-1} deg F^{-1}
152.8	5.9
-142.5	2.0
-142.8	0.62
- 53.5	0.71
7.9	0.75
52.0	0.82
153.3	2.5

Reference: NAS - 23

K+E 10 X 10 TO THE CENTIMETER 46 1515
18 X 25 CM. MADE IN U.S.A.
KEUFFEL & ESSER CO.

Figure 1. Thermal Conductivity of Polyurethane Foam (Density 1.8 lb ft^{-3}) in Vacuum

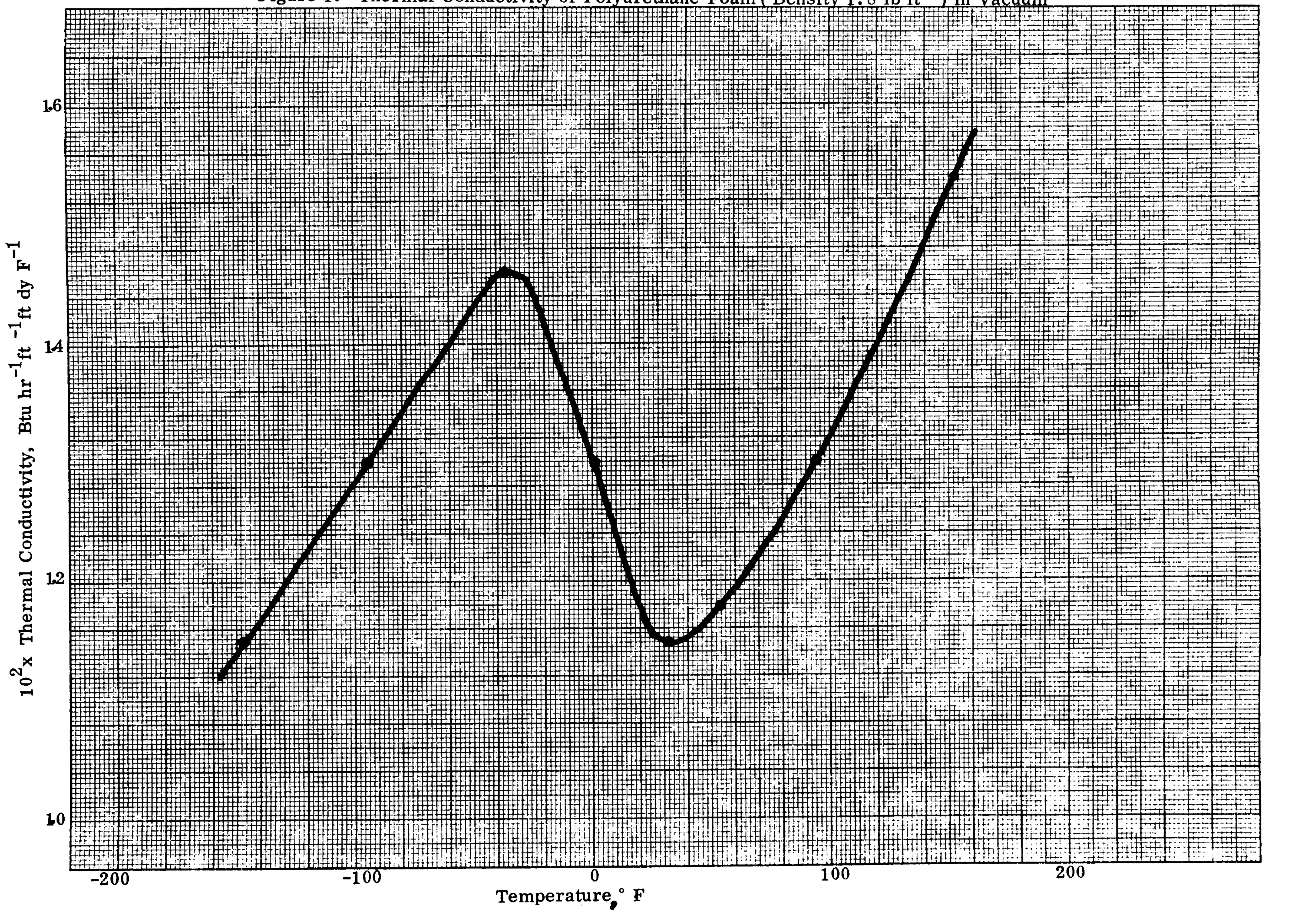

Figure 2. Thermal Conductivity of a Double Metallised Mylar-Foam Insulation In Vacuum