DETERMINATION OF THERMOPHYSICAL PROPERTIES OF ABLATIVE MATERIALS

PHASE I - LABORATORY DETERMINATIONS

Part B - Thermal Conductivity and Thermal Diffusivity

TRW SYSTEMS

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DETERMINATION OF THERMOPHYSICAL PROPERTIES OF ABLATIVE MATERIALS

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Part B - Thermal Conductivity and Thermal Diffusivity

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ABSTRACT

This report describes the work conducted under contract NAS9-4518 to perform laboratory measurements of thermophysical properties of three ablative materials being used on the Apollo Spacecraft primary propulsion systems. Two additional materials were also evaluated which have potential as improved ablative materials for Apollo applications. The thermophysical properties reported herein are comprised of thermal conductivity and thermal diffusivity. These properties were determined on Fiberite Corporation's MX 2600 and MX 2646, Western Backing Corporation's WBC 2234 and WBC 5217, and U. S. Polymeric Corporation's XR 2015. Description of the experimental techniques and test results are presented. A previous report of this program (issued 1 July 1966), covered laboratory measurements of heat capacity, density, melt and decomposition temperature on the same ablative materials.

SUMMARY

This report describes the work performed by TRW Systems for the National Aeronautics and Space Administration, Manned Spacecraft Center on Phase IB under Contract NAS9-4518. The primary objective of Phase I of this program was to perform laboratory measurements for the determination of thermophysical properties of selected virgin and charred ablative materials as a function of temperature. The thermophysical properties determined previously in Phase IA consisted of heat capacity, density, decomposition and melt temperature. The thermophysical properties determined in Phase IB, and reported here, comprise thermal conductivity and thermal diffusivity.

Thermal conductivity measurements were conducted as a function of temperature on both virgin and charred materials. Determinations covered a temperature range of 220°F to 450°F for the virgin materials and a range of 420°F to 1760°F for the charred specimens. Measurements were made using a specially designed twin guarded hot plate apparatus which was maintained in a vacuum environment. Several unique design features of the apparatus permitted measurements of temperature up to 1800°F.

The thermal diffusivity of both virgin and charred ablative materials was calculated as a function of temperature. The thermal diffusivity was calculated using thermal conductivity data reported in this report and heat capacity and density data reported previously in Phase IA of this program. Data for the virgin materials covered a temperature range of 200 to 400°F whereas the charred specimens covered a temperature range of 600°F to 1800°F.

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TABLES

ILLUSTRATIONS

1. INTRODUCTION

This report describes the second portion of the laboratory studies performed on The Study to Determine Thermophysical Properties of Ablative Materials for the National Aeronautics and Space Administration, Manned Spacecraft Center under Contract NAS9-4518. The primary objective of Phase I of this program was to perform laboratory measurements for the determination of thermophysical properties of selected virgin and charred ablative materials as a function of temperature. An earlier report (04812-6001-R000, Determination of Thermophysical Properties of Ablative Materials, Phase I, Part A, dated July 1, 1966) covered Part A of these laboratory determinations including:

- Heat capacity (both char and virgin)
- Density (both char and virgin)
- Decomposition temperature, and
- Melt temperature.

This report concludes the Phase I effort in presenting:

- Thermal conductivity (both charred and virgin), and
- Thermal diffusivity (both char and virgin)

properties as a function of temperature for each of the five materials investigated.

The data were determined for both virgin and charred materials up to 1800^{o}F. These data will be utilized for predictions of ablative material behavior in rocket engine environments. Under Phase II of this program, well-instrumented engine firings were conducted with a 1000-lb thrust rocket engine utilizing ablative thrust chambers made from the identical materials investigated in Phase I. Results of the ablative chamber behavior during the engine tests will be compared in the Phase II report with predicted values based on the thermophysical properties determined under Phase I of this effort.

The comparative results from laboratory measurements and engine measurements are expected to determine whether thermophysical property data obtained from controlled laboratory studies can be adequately extrapolated analytically to that observed in a dynamic engine firing. Furthermore, these data will improve significantly the capability of analytical predictions of ablative material behavior in rocket engine environments.

This report is organized into three principal sections. The first section describes the five ablative materials that were investigated. The next two cover the following thermophysical property measurements:

- Thermal conductivity
- Thermal diffusivity

In each of these sections, a detailed discussion is provided of the techniques employed, together with the results obtained for the five ablative materials.

2. MATERIALS INVESTIGATED

The thermophysical property measurements were conducted on the five ablative materials identified in Table 2.1.

TABLE 2.1
ABLATIVE MATERIALS INVESTIGATED

DESIGNATION	SUPPLIER	DESCRIPTION
MX 2600	Fiberite Corporation	Silica fabric and phenolic resin with a silica filler.
MX 2646	Fiberite Corporation	Silica fabric with a polyamide modified phenolic resin-no filler.
WBC 2234	Western Backing Corporation	"Irish Refrasil" with "high char" resin system.
WBC 5217	Western Backing Corporation	Magnesium hydroxide bulk fibers impregnated with a "high char" resin.
XR 2015	U.S. Polymeric Corporation	Silica fabric with elastomer modified, silica filled resin.

Further information concerning these materials is reported in the earlier Part A of this investigation (TRW Document 04812-6001-R000, Determination of Thermophysical Properties of Ablative Materials, Phase I, Part A, dated July 1, 1966).

3. THERMAL CONDUCTIVITY MEASUREMENTS

The thermal conductivity of the five virgin ablative materials and charred specimens was determined as a function of temperature. The experimental techniques employed and results obtained are presented in the following paragraphs.

3.1 EXPERIMENTAL TECHNIQUES

3.1.1 Thermal Conductivity Apparatus

The thermal conductivity of the ablative materials was determined by precise measurement of the temperature gradient across a specimen caused by a known heat flux. This measurement was accomplished using a twin guarded hot plate technique in an apparatus shown schematically in Figure 3.1. This apparatus consisted of a center heater, twin sample specimens and an upper and lower low temperature heater. The center heater was fabricated from Inconel and contained brazed heating elements. The heating element was a swaged magnesium oxide (MgO) resistance type element employing Nichrome V resistance wire. This resistance wire was encased in MgO insulation coaxially covered by an outer sheath of stainless steel. The outer diameter of the stainless steel-MgO-Nichrome V heating element was approximately 0.062 inches.

The guard ring heater, fabricated in a toroidal shape, was aligned with the center heater to insure that the heat generated in the center heater flows in only a direction parallel with the axis of the test specimens. The upper and lower heaters were adjusted at the identical preset temperature which was lower than that of the center heater to maintain a uniform temperature gradient across the specimen. The twin plate unit was thermally insulated from the surrounding environment by Min-K 2000 insulation. The insulated apparatus was contained within a steel vacuum vessel. During measurement of thermal conductivity the pressure in the system was between 0.010 and 0.15 torr.

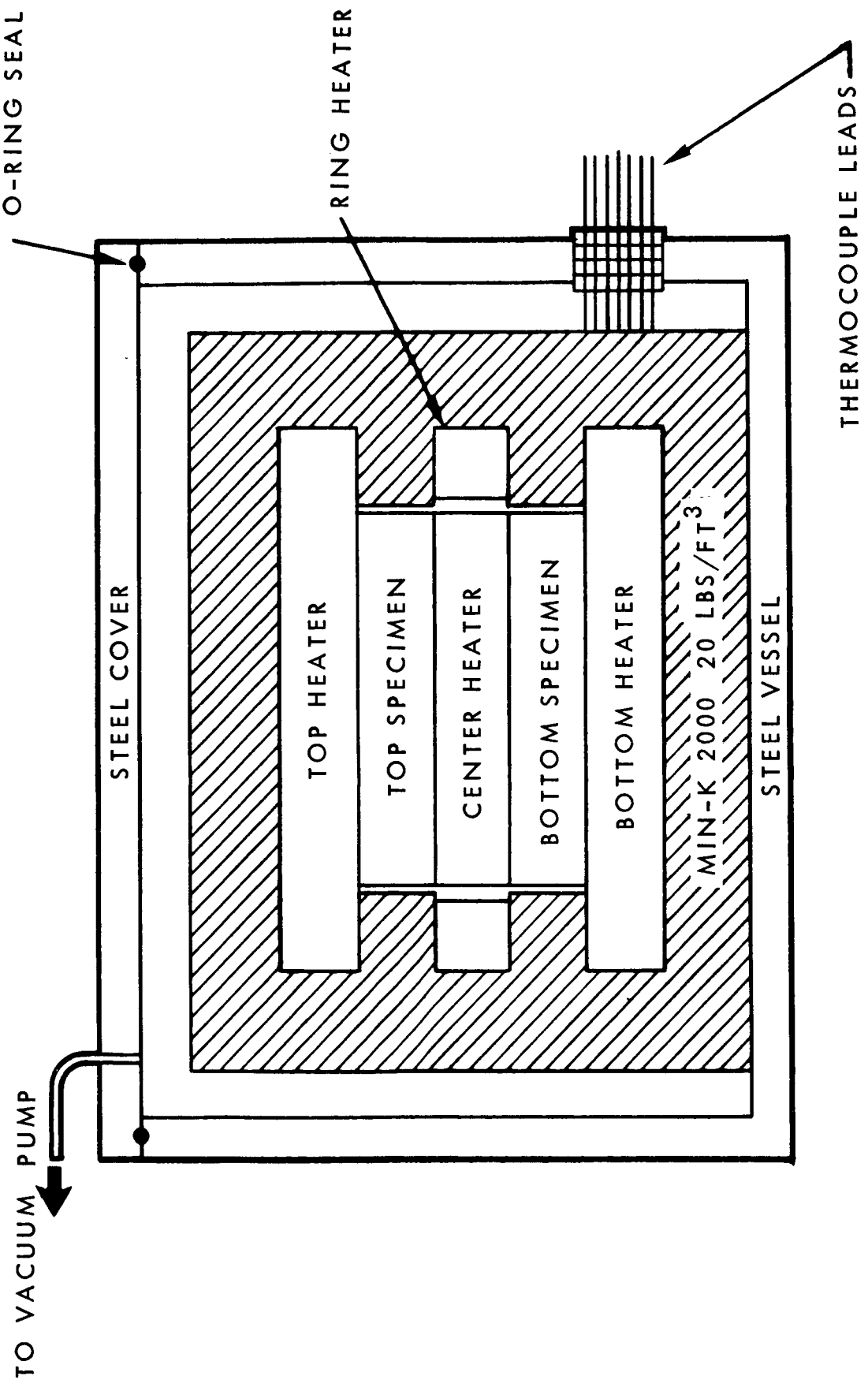


Figure 3.1. Schematic Drawing of the Thermal Conductivity Apparatus

The design of the apparatus was varied during the program to permit improved performance for the long term elevated temperature measurements (greater than 1200°F). Several design features which were implemented to permit measurements up to 1800°F were:

- Brazing the outer sheath of the heating element to the flat heater surfaces,
- Utilization of a single wire heating element,
- More effective insulation, and
- Aluminum foil reflectors between the steel chamber and the insulation.

A brief description of these design features and the advantages they afforded is presented below.

Brazing the outer sheath of the heating element to the flat heater surfaces provided a direct conductive means for transfer of the heat generated in the heating element to the surfaces of the heaters. The conductive mode of heat transfer was required in the heating element; otherwise the vacuum in the system would only permit radiative transfer. During some of the early work at specimen temperature of around 1400°F, it was found that the 347 stainless steel sheath melted when brazing was not employed. The melting point of 347 stainless steel occurs at approximately 2550°F to 2600°F. By brazing the element to the sheath, conduction rapidly transferred the heat generated to the Inconel body and a lower sheath operating temperature results.

Conversion from two wire elements to a single wire element eliminated a problem of leads shorting out. The two wire elements had a small spacing between the Nichrome wires between which shorting frequently occurred because of the differential expansion characteristic encountered when the element reached operating temperature. The single wire design overcame this problem.

The total heat requirement of the system was reduced further by utilizing a more effective insulation surrounding the twin plate unit.

Initially, a Dynaquartz insulation material was used. This was changed later to Min-K 2000 of 20 pounds per cubic foot density. The Min-K 2000 was a much more effective insulation system and thus reduced the total power required.

The addition of aluminum foils between the steel chamber and the Min-K insulation also help to reduce the power requirement. Several layers of aluminum foil were placed in this annulus to act as radiation barriers. The surface area of heating element available was increased by providing for more total element length within the Inconel body. These changes together with the brazing of the element enabled measurements to be made at the higher mean temperatures.

It was originally planned that 1800°F mean temperature measurement would be obtained. This 1800°F temperature had to be reduced, however, when it was noted that the brazing alloy had partially melted after exposure to approximately 1800°F during measurement of the MX 2646 charred material.

Specimens of the ablative material under investigation were placed on each side of the center heater. These specimens were 2.50 inches in diameter and 0.50-inch thick. Early in the program the temperature across the specimens was assumed to be equal to the temperatures measured in the Inconel center heater and top and bottom heater. This method assumed that there was no significant temperature drop between the heater and the surface of the test specimens. An investigation to confirm the validity of this assumption was made and it was found that an appreciable temperature gradient existed. The magnitude of this contact resistance was very significant in determination of the thermal conductivity value. For example, in one case, a temperature gradient of approximately 10°C existed on each side of the specimen when a total temperature gradient of 40°C was imposed between the heater surfaces. This gradient was determined by direct measurement of the specimen surfaces by use of thermocouples laminated to the specimen. Two plies of MX 2600 fabric were used to effect this laminar bond.

When this relationship was established it was decided to repeat all earlier measurements which were not made with bonded thermocouples.

These bonded thermocouples effectively overcame contact resistance resulting in higher thermal conductivity values than would be obtained with unbonded thermocouples.

The top and bottom heaters were used infrequently during the program as a source of additional power when virgin materials were measured. When charred materials were measured at high mean temperatures the top and bottom heaters were frequently employed to reduce the overall wattage requirement of the system. This effectively lead to longer heating element life for the center heater.

The steel chamber surrounding the apparatus was essentially a pressure vessel to enable measurements to be made under vacuum. Measurement under vacuum conditions was required

- to prevent oxidation of the virgin and charred materials at high mean temperatures, and,
- to reduce the total power requirement of the system.

This power reduction was possible because the Min-K insulation is much more effective under vacuum conditions; the aluminum foil radiation barriers were also used to reduce heat transfer to the steel pressure vessel. A removable cover with a silicone rubber O-ring permitted the apparatus to be disassembled for removal and installation of samples.

The guard ring heater surrounding the center heater effectively isolated the center heater from radial heat flow. This was accomplished by maintaining the guard ring heater at the same temperature as the center heater. When these two temperatures were the same, no heat should flow between the center heater and the guard ring heater. Thus all wattage imposed on the center heater effectively was used in heating the two specimens of material being tested.

3.1.2 Preparation of Test Specimens

The thermal conductivity test specimens, with the exception of the WBC 5217 material, were molded with a ply orientation of 45° to the laminate surface into cylinders having dimensions 2.50 inch diameter x 0.500 inch high. The WBC 5217 material was molded with a random orientation although the direction of applied pressure was perpendicular to the laminate surface. Weight losses of the specimens after ignition in air at 1800°F were reported under the Part A portion of the Phase I investigation. All of the materials investigated have found application as ablative materials in liquid or solid rocket engine applications.

The swaged MgO thermocouple was laminated to the surface of the test specimen using two plies of MX 2600 phenolic preimpregnated cloth. The orientation of the thermocouples on the surface of the test specimen is shown in Figure 3.2.

The charred materials were prepared by heating the virgin materials in a closed container in a nitrogen atmosphere. The samples were supported off the bottom of the container to prevent contamination from the liquid decomposition products that were generated during pyrolysis. A heating cycle of two hours at each of the following temperatures was used, 392°F, 752°F, 1112°F, and 1472°F. It was determined that the WBC 5217 material developed cracks and excessive shrinkage occurred during carbonization. For this reason it was not possible to determine the thermal conductivity of this material.

Thermocouples were laminated to the charred specimen in a manner similar to that utilized for the virgin materials. The two-ply glass reinforced phenolic laminate covering of the thermocouple was charred subsequently using the same heating cycle as that employed to char the test specimen.

3.1.3 Operational Procedure

The operational procedure employed in obtaining the thermal conductivity measurements consisted of assembly of the test specimens

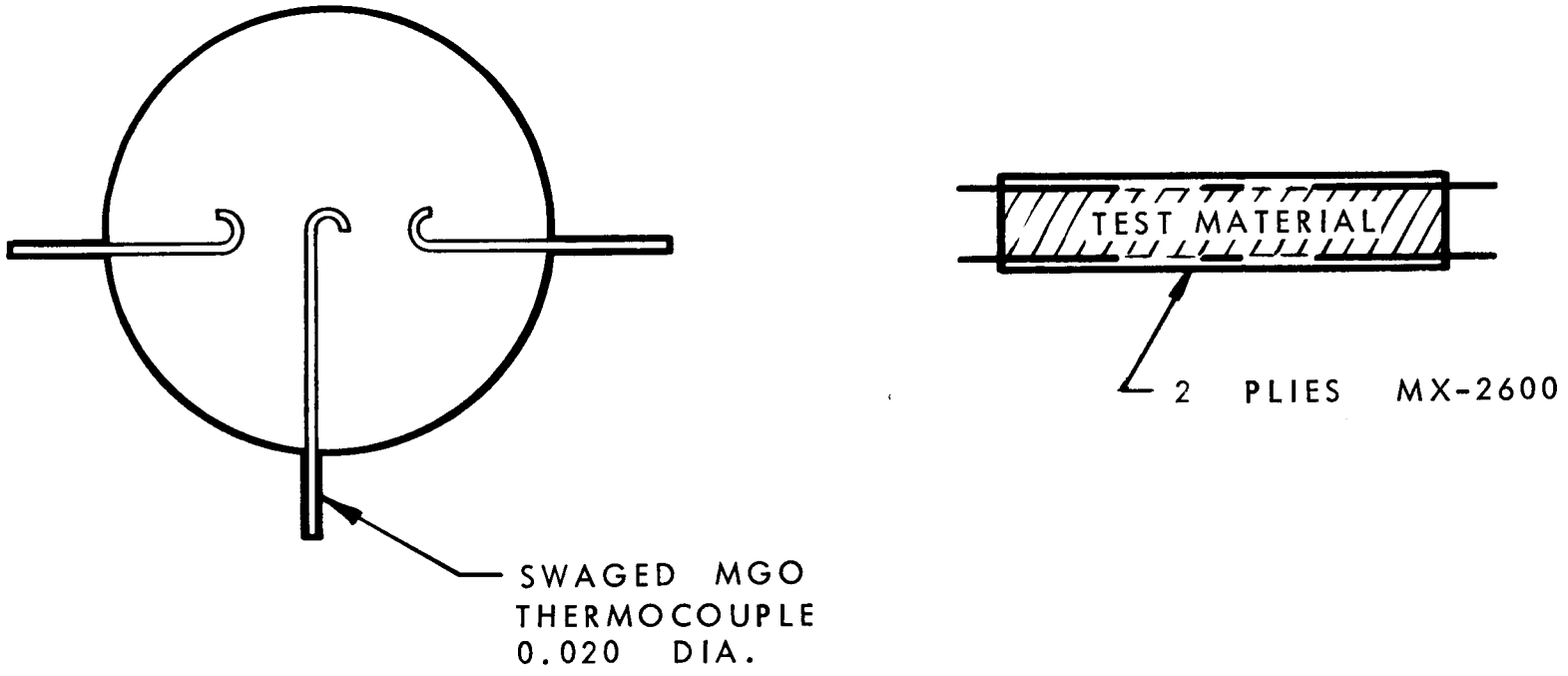


Figure 3.2. Specimen-Thermocouple Configuration

between the bottom heater, center heater and top heater, insertion of the Min-K 2000 insulation, closing the lid and evacuation of the system to a pressure between 0.010 and 0.150 torr. When the vacuum had been attained the heaters were activated and adjusted to levels which would provide a desired specimen mean temperature.

A minimum time of 24 hours was required for the apparatus to attain equilibrium. Normally most of the temperature adjustments during this period were required to equalize the temperature between the guard ring and the center heater. Some adjustments were necessary to the top and bottom heater when charred materials were run at high mean temperature.

Figure 3.3 illustrates data taken for a typical conductivity determination. The apparatus is shown schematically in this figure with appropriate blanks for insertion of millivolts readings obtained from the thermocouples employed. These millivolts readings were reduced to temperatures and appropriate mean temperatures and ΔT or temperature differential between the top and bottom surfaces of the specimens were determined for both the top and bottom specimens. The average mean between the top and bottom specimens was reported as the specimen temperature and it was assumed that the heat flow to the top and bottom specimens was proportional to the ΔT's.

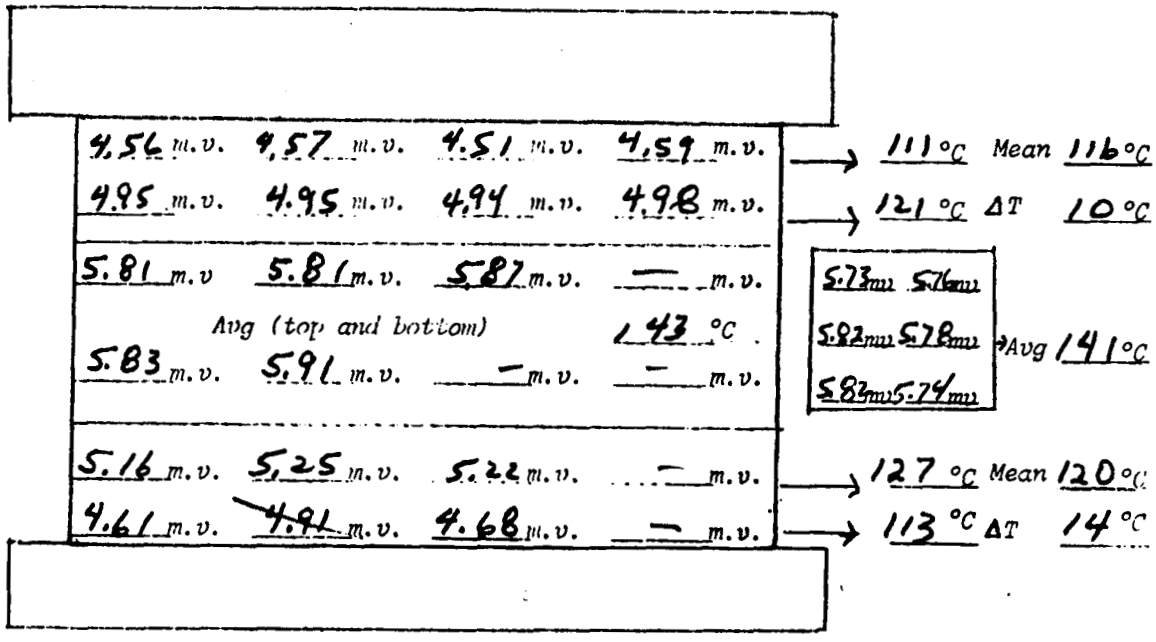
The thermal conductivity was calculated as follows:

$$k = \frac{Eil}{\phi A \Delta T} \qquad (1)$$

E = voltage applied to the center heater
i = current flow in center heater
l = sample thickness
ϕ = mechanical equivalent of heat
A = sample area
ΔT = temperature difference across sample surface

THERMAL CONDUCTIVITY DETERMINATION

(All Thermocouples Chromel-Alumel)



Material MX 2600 VIRGIN Thickness 0.500.

Orientation to flat surface 45°.

Date reading obtained 9-24-66. Time 2:04.

Intended mean temperature 121 °C.

Actual mean temperature 118 °C, 244.4 °F.

Center Heater: Volts: 5.15 Amps: 0.96.

Guard Heater: Volts: 4.9 Amps: —.

Top Heater: Volts: 0 Amps: 0.

Bottom Heater: Volts: 0 Amps: 0.

Thermal Conductivity = $9.60 \times 10^{-3} \frac{\text{Power Center Heater}}{\Delta T \text{ Top} + \Delta T \text{ Bottom}}$ (For 0.500" thick sample)

$= 1.975 \times 10^{-3}$ Cal/Cm sec °C

$= 1.975 \times 10^{-3} \times 2903 = 5.73$ BTU-in/hr ft^2 °F

REMARKS:

Figure 3.3. Typical Data Summary Sheet for Thermal Conductivity Determination

Expressing equation (1) in English units and utilizing the dimensions of the test specimens employed in this work, the following equation is derived:

$$k = 27.3 \frac{Ei}{\Delta T_1 + \Delta T_2} \qquad (2)$$

where:

k = thermal conductivity, BTU-inch/hr-ft^2-$^\circ$F
E = voltage applied to center heater, volts
i = current flow center heater, amps
ΔT_1 = temperature difference across the top specimen, $^\circ$C
ΔT_2 = temperature difference across bottom specimen, $^\circ$C

The thermal conductivity values obtained using equation (2) can be converted to C.G.S. units (cal/cm-$^\circ$C-sec) by dividing by 2903.

3.2 EXPERIMENTAL RESULTS

3.2.1 Virgin Materials

The results of the thermal conductivity measurements on the virgin materials as a function of temperature are shown in Figures 3.4 through 3.8. These values are, in general, somewhat higher than other measurements reported in the literature. It is believed that the primary reason for this is that bonded thermocouples were employed for this study.

Among the virgin materials evaluated, the polyamide modified MX 2646 appeared to have the lowest conductivity. The elastomer modified XR 2015 was only slightly higher at comparable mean temperatures. A comparatively low value was obtained for the WBC 2234 at a mean temperature of 221°F but the conductivity at 480°F was considerably higher. The MX 2600 material was higher than the other silica reinforced materials. All of the silica reinforced materials showed an increasing conductivity with increasing mean temperatures.

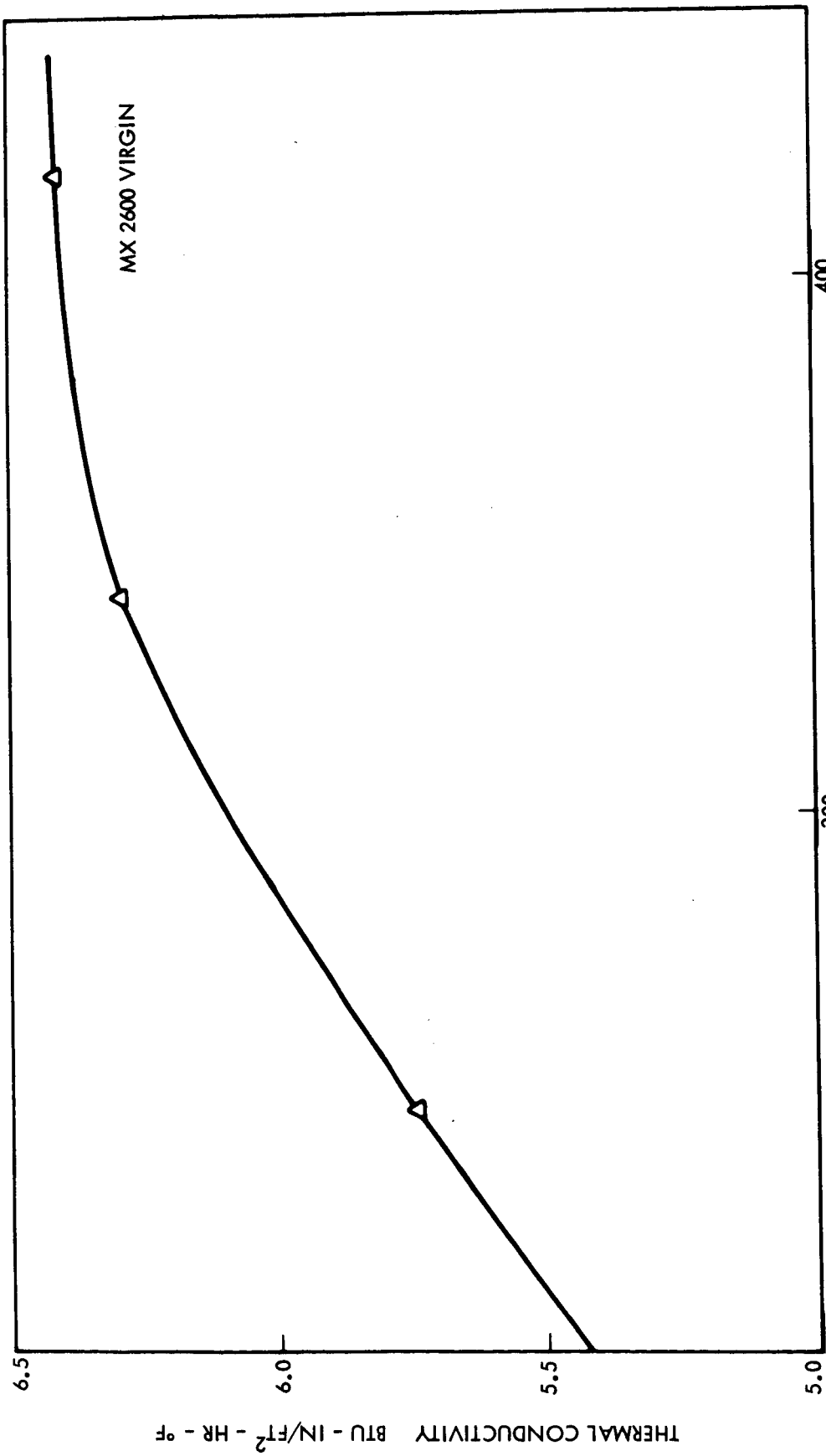


Figure 3.4. Thermal Conductivity of Virgin MX 2600 as a Function of Temperature

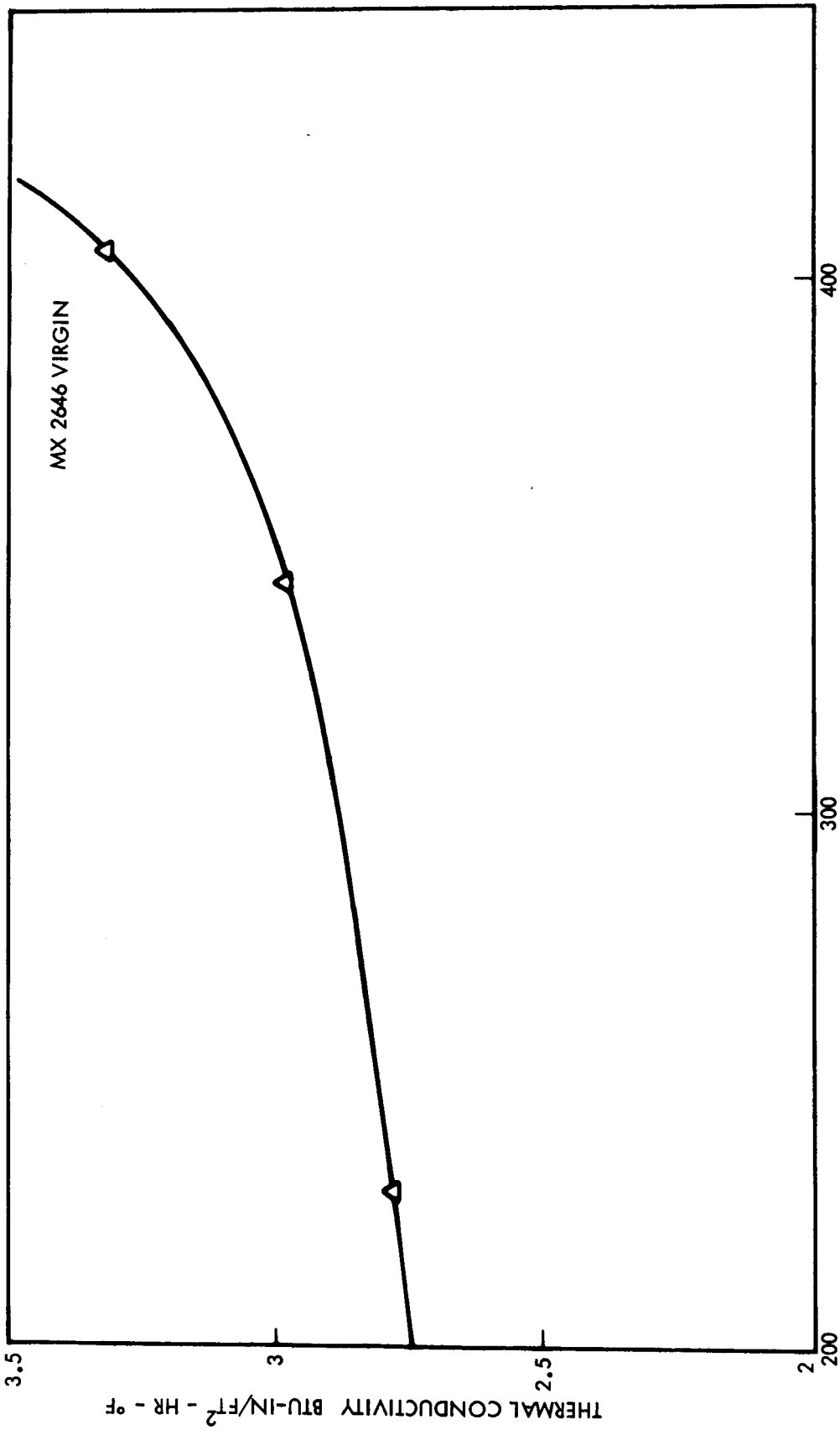


Figure 3.5. Thermal Conductivity of Virgin MX 2646 as a Function of Temperature

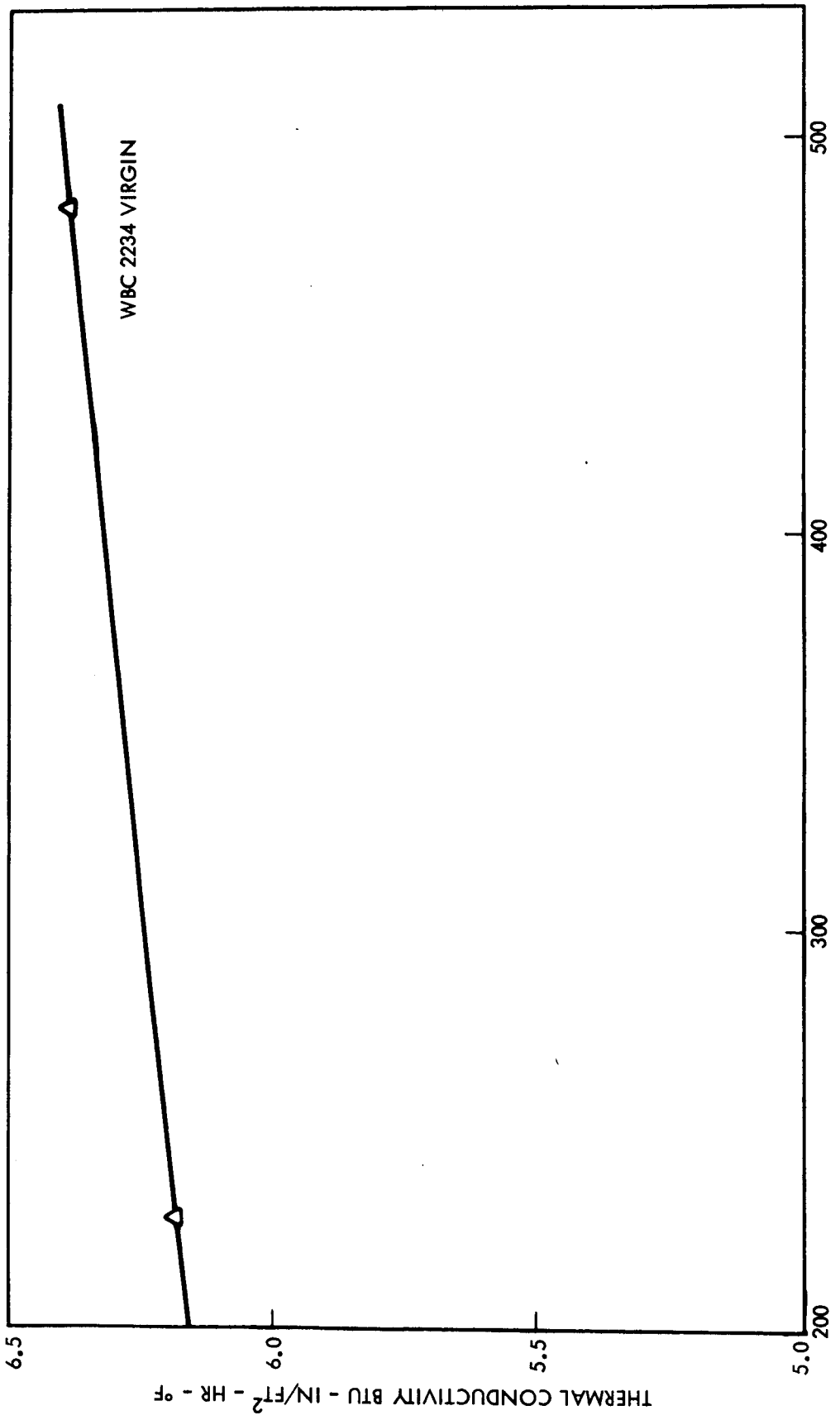


Figure 3.6. Thermal Conductivity of Virgin WBC 2234 as a Function of Temperature

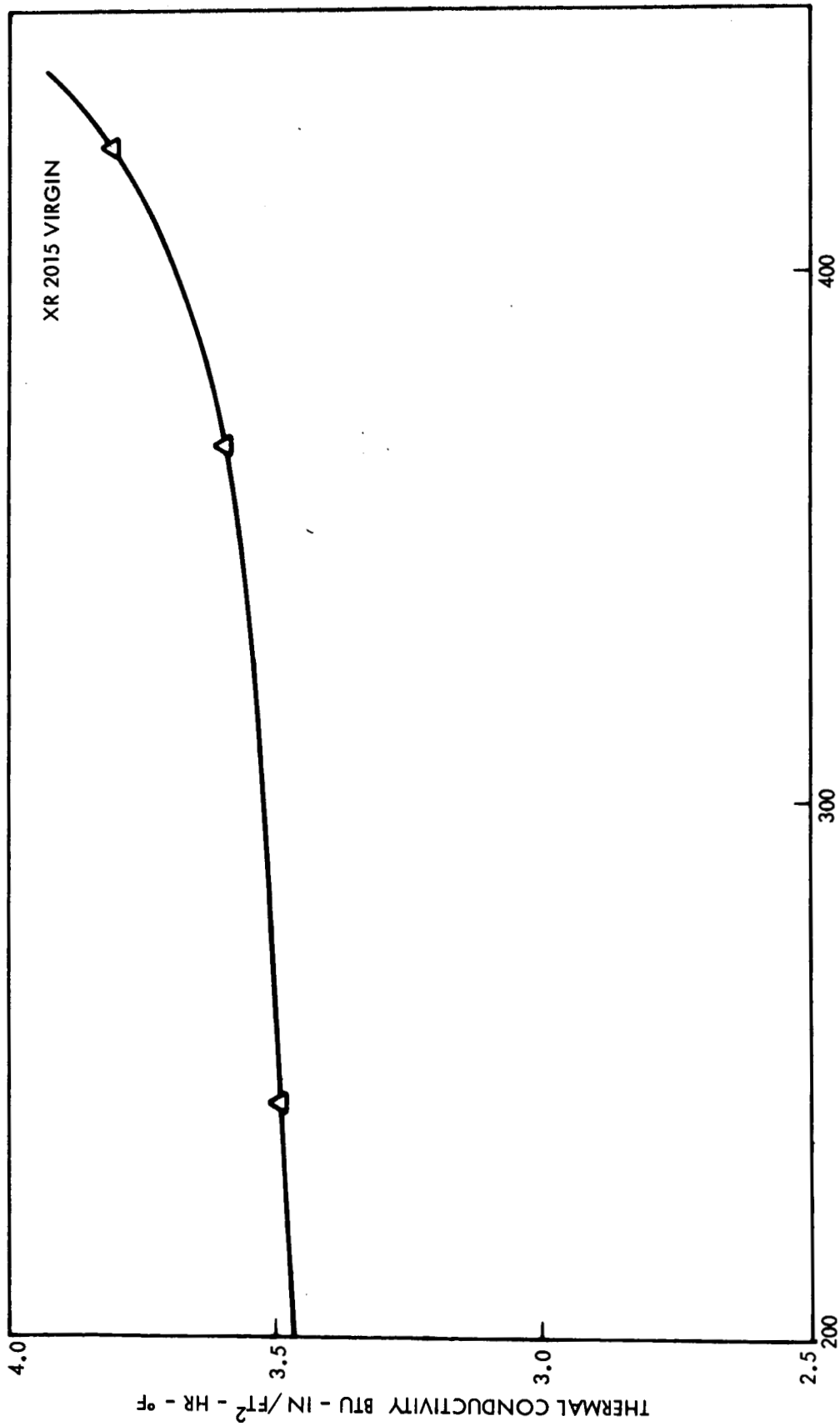


Figure 3.7. Thermal Conductivity of Virgin XR 2015 as a Function of Temperature

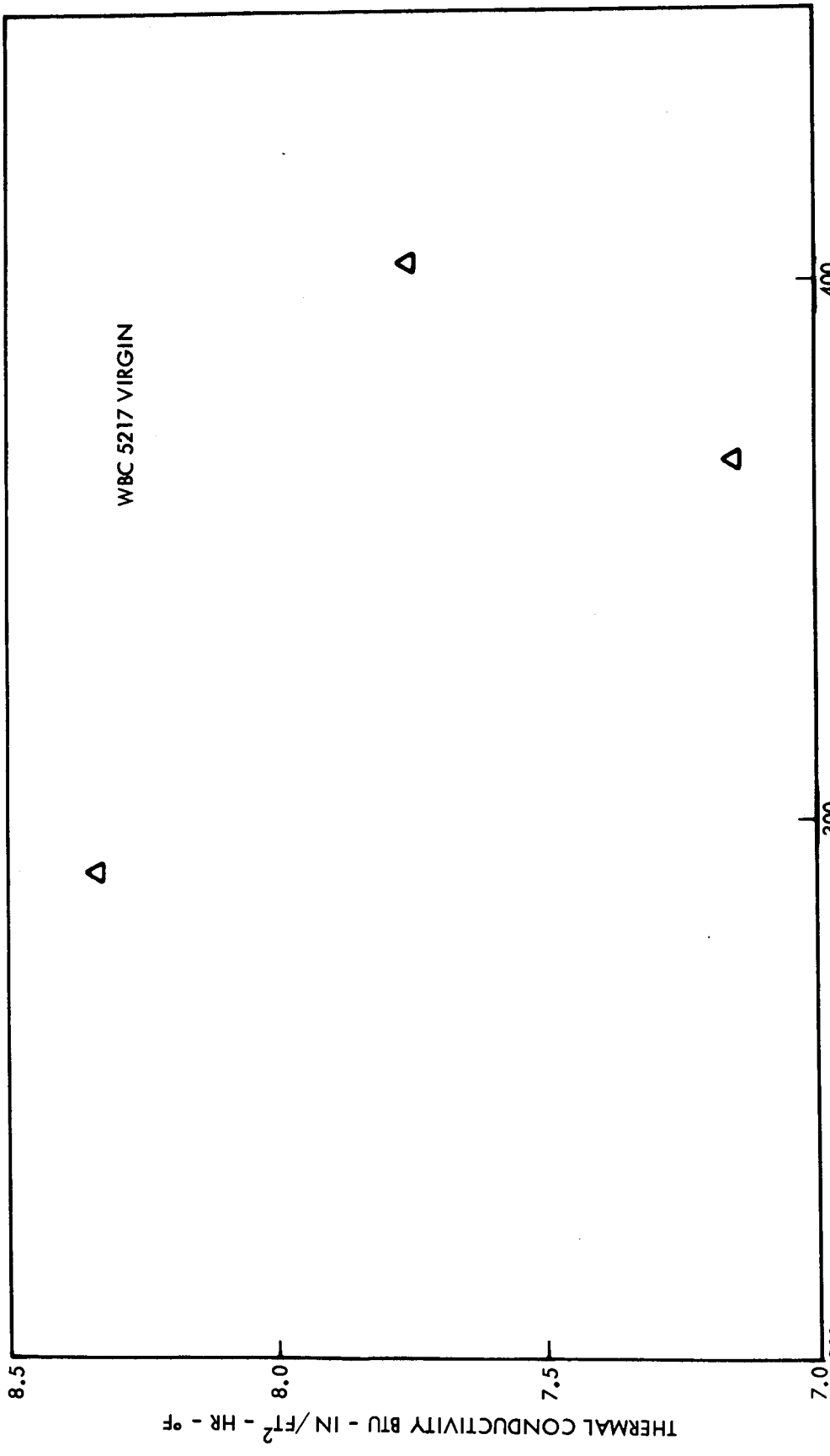


Figure 3.8. Thermal Conductivity of Virgin WBC 5217 as a Function of Temperature

The magnesium hydroxide reinforced WBC 5217 gave scattered thermal conductivity values as a function of temperature. Consequently, the data points shown in Figure 3.8 were not connected by a smooth curve.

3.2.2 Charred Specimens

The results of the thermal conductivity measurements on the charred specimens as a function of temperature are shown in Figures 3.9 through 3.12. It is seen that all of the charred materials exhibited an increase in thermal conductivity with mean temperature. It is notable that very high conductivity values were obtained at mean temperatures above 1400°F for all of the materials evaluated. It was not possible to determine thermal conductivity on the charred WBC 5217 material because dimensional integrity was not maintained and cracks developed during the charring process.

It should also be mentioned that these values were obtained under vacuum conditions and while it is not felt that this should materially change measured properties of virgin materials, the measurement of charred materials might be more significantly effected because of the porous nature of the char. However the variation which might be expected because of operation in a vacuum was considerably less than that which would have been caused by operation in air (oxidation of product) or in an inert gas (greater heat losses and hence lower mean temperatures and burnt out heating elements).

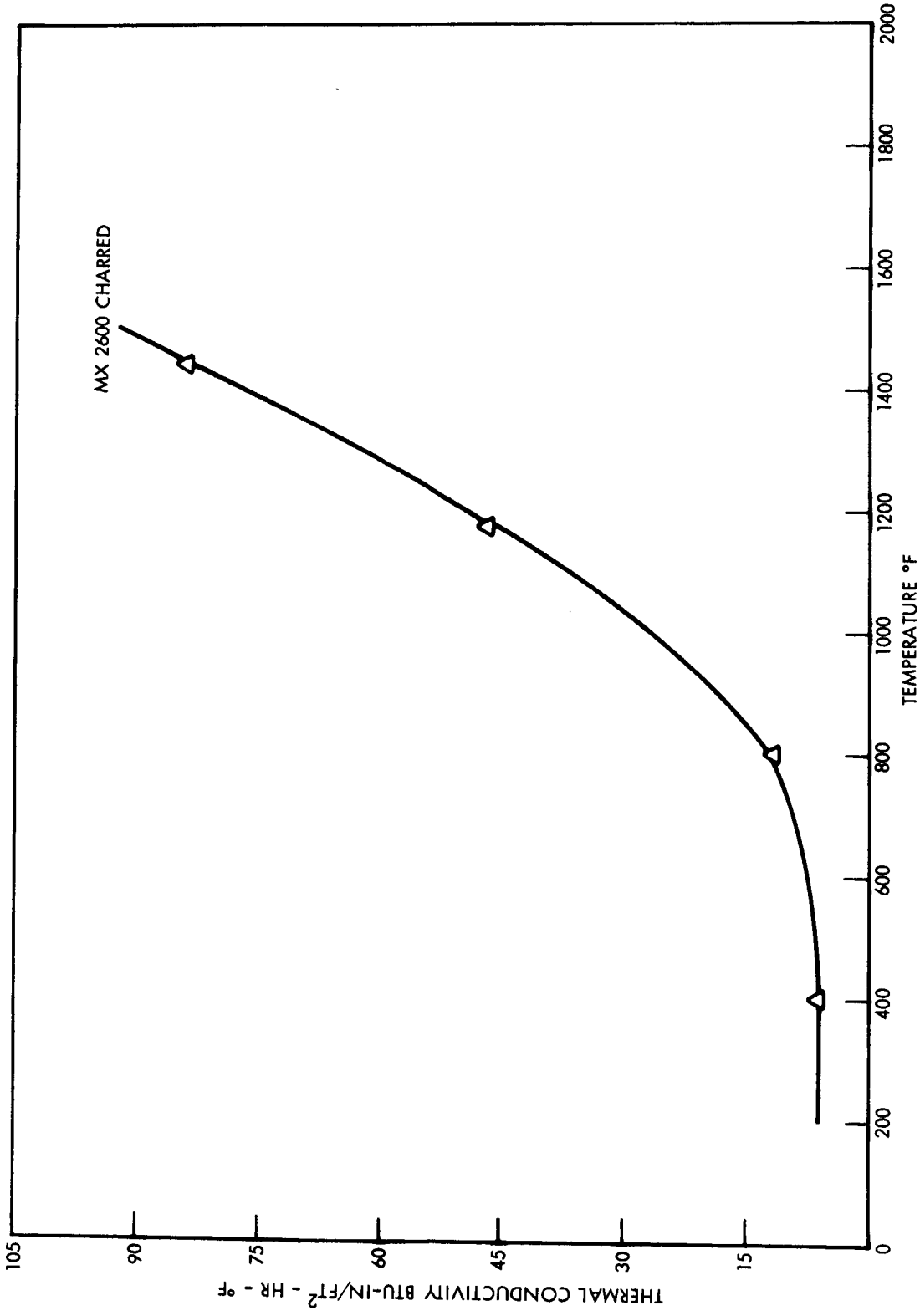


Figure 3.9. Thermal Conductivity of Charred MX 2600 as a Function of Temperature

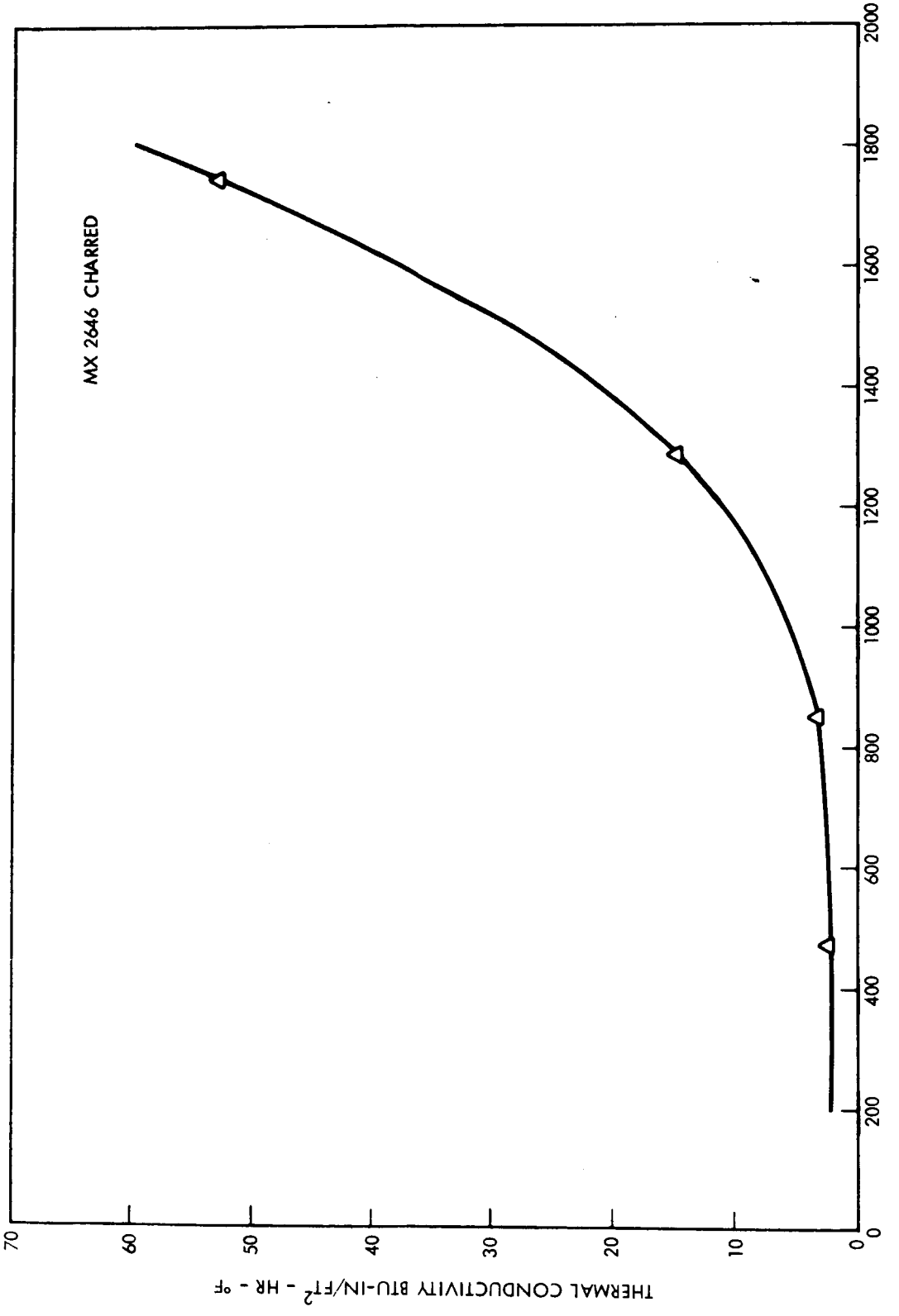


Figure 3.10. Thermal Conductivity of Charred MX 2646 as a Function of Temperature

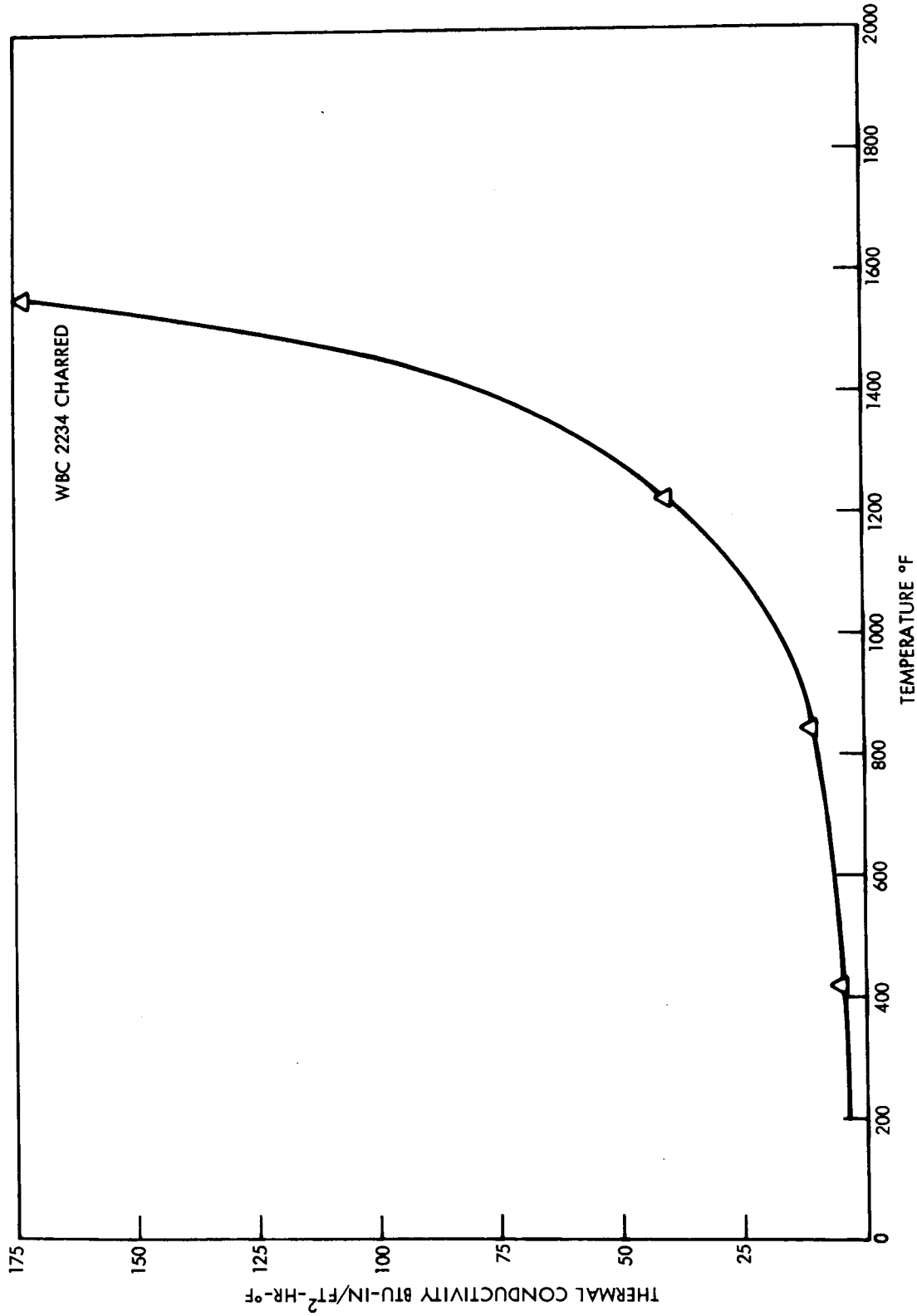


Figure 3.11. Thermal Conductivity of Charred XR 2015 as a Function of Temperature

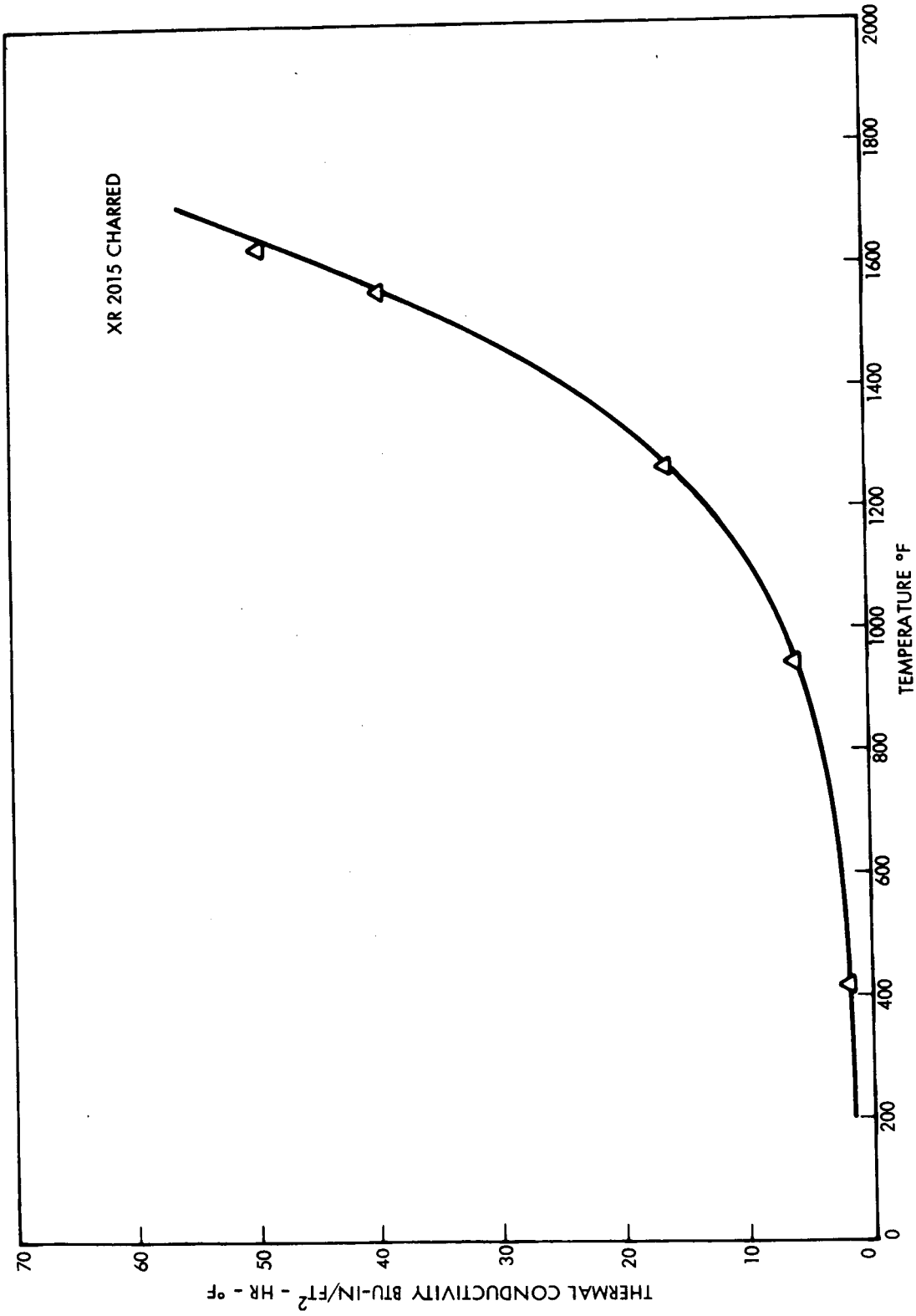


Figure 3.12. Thermal Conductivity of Charred XR 2015 as a Function of Temperature

4. THERMAL DIFFUSIVITY CALCULATIONS

4.1 PROCEDURE

Thermal diffusivities were calculated for the virgin and charred ablative materials as a function of temperature. This calculation was performed using equation 3.

$$\alpha = \frac{1}{12} \frac{k}{\rho Cp} \tag{3}$$

Where:

α = thermal diffusivity, $ft^2/hr.$
k = thermal conductivity, $\frac{BTU\text{-}in}{hr\text{-}ft^2\text{-}^oF}$
ρ = density, lb/ft^3
Cp = heat capacity, $BTU/lb\text{-}^oF$

The values of thermal conductivity, density and heat capacity used in calculation of thermal diffusivity were obtained from smoothed temperature dependent curves shown in Section 3.2 of this report and in Part A of the Phase I laboratory study. Because the density-temperature relationship for the charred materials was nearly linear it was possible to extrapolate densities with confidence below 1000^oF to at least 600^oF.

4.2 RESULTS

The thermal diffusivity of the virgin and charred materials were calculated using equation 3. The results of calculations on the virgin materials are presented in Table 4.1 together with a summary of thermal conductivities, heat capacities and densities used for the calculation. The virgin material thermal diffusivities are plotted as a function of temperature between 200 and 400^oF in Figure 4.1.

The calculated thermal diffusivities of the charred specimens, together with a summary of the thermophysical properties required for

TABLE 4.1

SUMMARY OF THERMOPHYSICAL PROPERTIES OF VIRGIN MATERIALS AS A FUNCTION OF TEMPERATURE

Material	Temperature ^{o}F	Thermal Conductivity BTU-in/ft^2-hr-^{o}F	Heat Capacity BTU/lb-^{o}F	Density lb/ft^3	Thermal Diffusivity ft^2/hr
MX 2600	200	5.43	0.27	107.0	0.0156
	300	6.09	0.28	106.0	0.0170
	400	6.40	0.29	104.0	0.0176
MX 2646	200	2.75	0.26	111.5	0.0079
	300	2.88	0.27	110.3	0.0080
	400	3.28	0.29	108.7	0.0086
WBC 2234	200	6.16	0.26	106.5	0.0185
	300	6.24	0.27	104.0	0.0185
	400	6.32	0.28	101.0	0.0185
XR 2015	200	3.47	0.30	90.0	0.0107
	300	3.53	0.32	88.0	0.0104
	400	3.68	0.35	84.0	0.0104
WBC 5217	200	--	0.35	117.0	--
	300	8.35	0.38	115.5	0.0158
	400	--	0.41	113.5	--

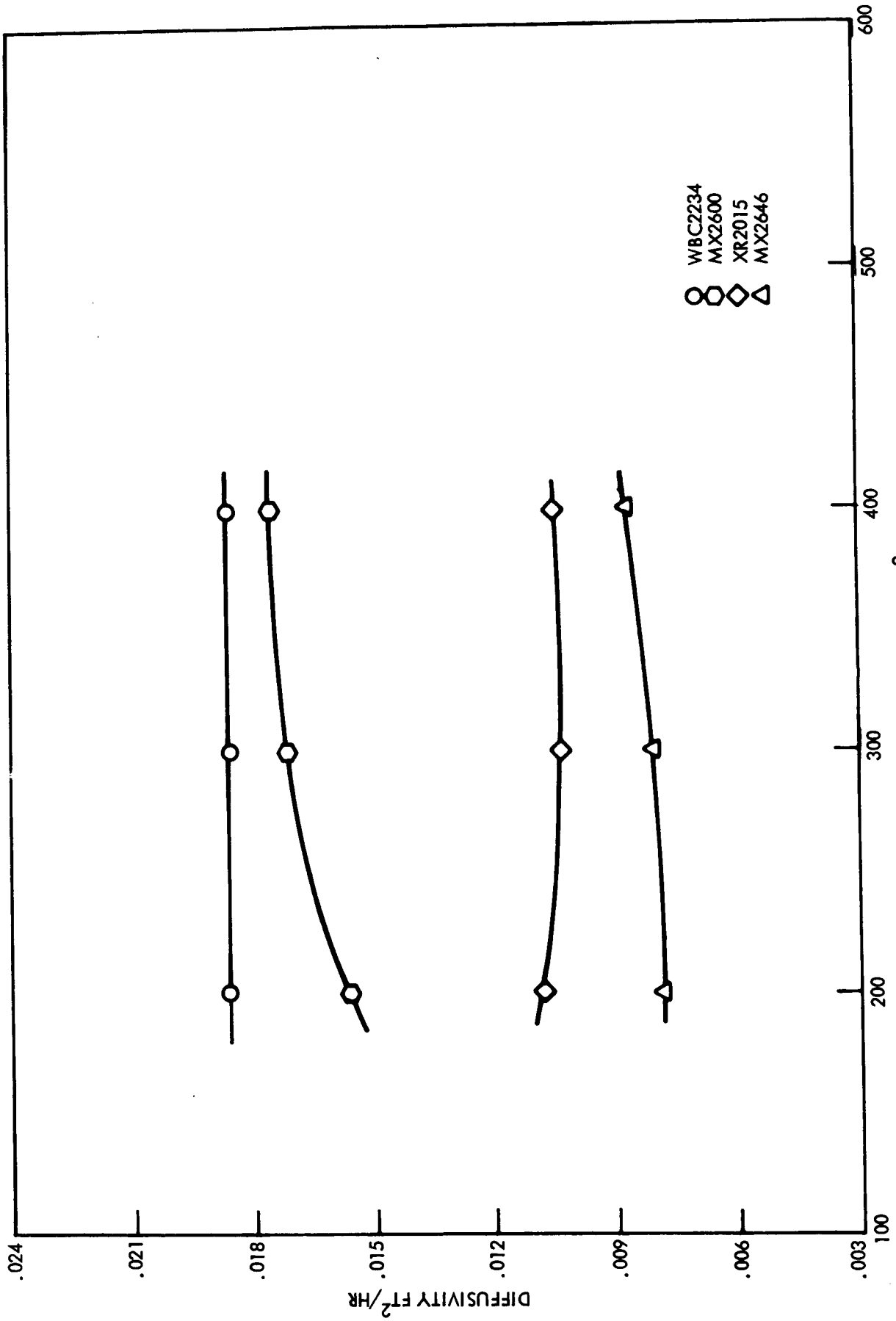


Figure 4.1. Thermal Diffusivity of Virgin Materials as a Function of Temperature

TABLE 4.2

SUMMARY OF THERMOPHYSICAL PROPERTIES OF CHARRED MX 2600 AS A FUNCTION OF TEMPERATURE

Temperature ^{o}F	Thermal Conductivity BTU-in/ft^2-hr-^{o}F	Heat Capacity BTU/lb-^{o}F	Density lb/ft^3	Thermal Diffusivity ft^2/hr
600	7.5	0.27	94.0[a]	0.025
800	12.0	0.31	94.0[a]	0.034
1000	27.0	0.33	94.0	0.073
1200	49.5	0.34	94.0	0.129
1400	78.5	0.35	94.0	0.199
1600	108.[a]	0.35	94.0	0.274
1800	139.[a]	0.35	94.0	0.35

[a]Extrapolated Value

TABLE 4.3

SUMMARY OF THERMOPHYSICAL PROPERTIES OF CHARRED MX 2646 AS A FUNCTION OF TEMPERATURE

Temperature ^{o}F	Thermal Conductivity BTU-in/ft^2-hr-^{o}F	Heat Capacity BTU/lb-^{o}F	Density lb/ft^3	Thermal Diffusivity ft^2/hr
600	2.2	0.24	96.0[a]	0.0079
800	3.0	0.27	95.0[a]	0.0097
1000	5.4	0.28	94.0	0.0170
1200	11.2	0.28	93.0	0.035
1400	21.3	0.28	92.0	0.069
1000	37.9	0.28	91.0	0.124
1800	59.2[a]	0.28	90.0	0.196

[a]Extrapolated Value

TABLE 4.4

SUMMARY OF THERMOPHYSICAL PROPERTIES OF CHARRED WBC 2234 AS A FUNCTION OF TEMPERATURE

Temperature ^{o}F	Thermal Conductivity BTU-in/ft^2-hr-^{o}F	Heat Capacity BTU/lb-^{o}F	Density lb/ft^3	Thermal Diffusivity ft^2/hr
600	6.5	0.27	94.0[a]	0.021
800	9.6	0.28	93.0[a]	0.031
1000	17.5	0.30	92.5	0.053
1200	37.0	0.31	92.0	0.108
1400	78.	0.31	91.0	0.23
1600	218.[a]	0.31	90.0	0.65

[a]Extrapolated Value

TABLE 4.5

SUMMARY OF THERMOPHYSICAL PROPERTIES OF CHARRED XR 2015 AS A FUNCTION OF TEMPERATURE

Temperature ^{o}F	Thermal Conductivity BTU-in/ft^2-hr-^{o}F	Heat Capacity BTU/lb-^{o}F	Density lb/ft^3	Thermal Diffusivity ft^2/hr
600	2.4	0.25	64.0[a]	0.0125
800	4.1	0.27	64.0[a]	0.0197
1000	6.8	0.28	64.0	0.032
1200	12.8	0.29	64.0	0.057
1400	24.0	0.30	64.0	0.104
1600	43.3	0.30	64.0	0.188
1800	70.[a]	0.30	64.0	0.30

[a]Extrapolated Value

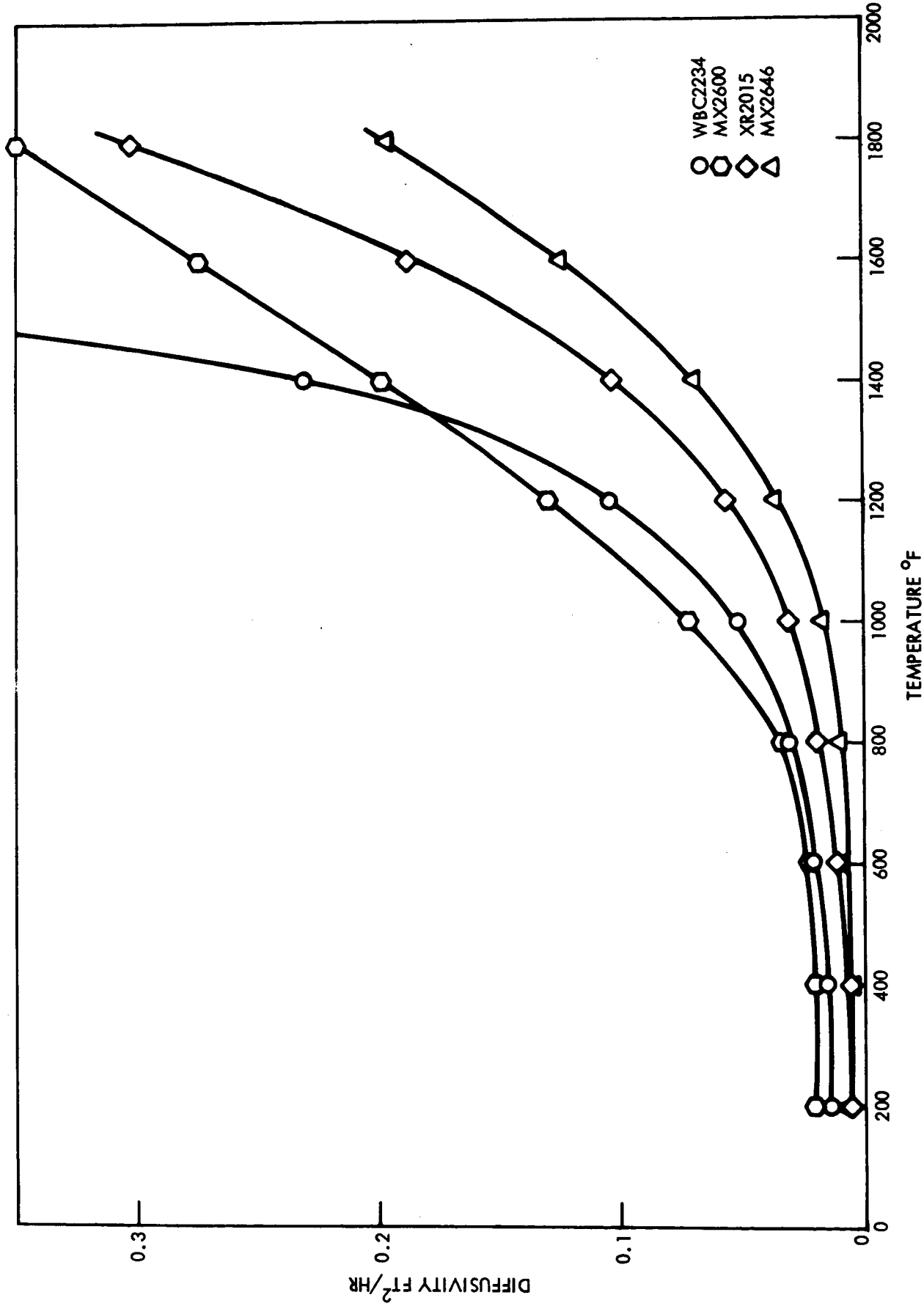


Figure 4.2. Thermal Diffusivity of Charred Specimens as a Function of Temperature

their calculation, are listed in Tables 4.2 through 4.5. Plots of the charred specimens thermal diffusivities as a function of temperature are presented in Figure 4.2.

The most significant factor affecting the thermal diffusivity over the temperature range evaluated is the thermal conductivity variations that were observed. Neither heat capacity nor density variations were as significant on a percentage variation basis as the thermal conductivity. The greatest change in thermal diffusivity was observed for the charred materials in comparing values determined at low mean temperatures with those calculated at high mean temperatures.