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VACUUM THERMAL CONDUCTIVITY MEASUREMENTS OF NASA E4A1 ELASTOMERIC HEAT SHIELD MATERIAL

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Abstract

Vacuum thermal conductivity measurements utilizing the line source technique were made for NASA E4A1 elastomeric heat shield material. Thermal conductivity measurements were made on both vacuum exposed and ambient samples to discern the changes due to vacuum exposure. Measurements were also made on the vacuum samples after reexposure to the atmosphere. The results indicated that vacuum exposure caused about a fifteen per cent reduction in thermal conductivity during the 9-day vacuum exposure period. On reexposure to the atmosphere, the thermal conductivity reverted to its preexposure value.

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Nomenclature

c	Specific heat, B/lbm F
I	Current, amperes
K	Thermal conductivity, B/hr ft F
P	Absolute pressure, Torr
q	Heat generated per unit length, B/hr ft
r	Radial distance from heater wire, ft
R	Resistance of heater wire, ohms/ft
s	Strength per unit length of a continuous line source, F ft^2/hr
S	Strength of an instantaneous point source, F ft^3
t	Time, seconds
t_o	Time correction factor, seconds
v	Dummy variable
x,y,z	Cartesian coordinates
α	Thermal diffusivity, ft^2/hr
γ	Constant
ρ	Density, lbm/ft^3

Subscripts are used to indicate the value of a variable at a particular time. For example, θ_1 is the value of θ when $t = t_1$.

Special symbols used in some of the calculations given in the appendix are defined when used and do not appear elsewhere in the report.

The term "run number" is used to record experimental data. The time interval between runs was 24 hours. Run number 1 includes the initial data for all samples. For runs 2 through 10, the vacuum system and humidity chamber were in operation. For runs 11 through 13, all samples were exposed to the atmosphere.

Introduction

The recent work by Greenwood(1)* concerning vacuum induced changes in the mechanical properties of an elastomeric material has emphasized the need for measuring the properties of certain organic materials while they are exposed to a vacuum.

The purpose of this investigation was to develop a technique for measuring the thermal conductivity of a heat-shield material during vacuum exposure and to conduct exploratory measurements to determine the extent of changes compared to the values at atmospheric conditions.

The line source method of measuring thermal conductivity appeared to be the most promising technique to develop because it would provide a means for making the necessary measurements while permitting maximum exposure of the sample to the vacuum. Also, since no outside covering would be required, the method would also be suitable for making measurements in any desired atmosphere. An additional advantage is the simplification of the vacuum feedthrough problem because a heat sink is not required such as would be necessary with a hot plate method.

Explanation of the Line Source Method

The line source method for measuring thermal conductivity has been used by many investigators and it is no longer clear who originated it. The first comprehensive evaluation of the method was given by Van der Held and Van Drunen(2) in 1949.

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*Superscript numbers refer to items in the bibliography.

The governing equations for the analysis of a line source can be deduced from the book by Carslaw and Jaeger(3) and the following has been adapted from reference 3.

The differential equation for heat conduction can be written as

$$\frac{\partial^2\theta}{\partial x^2} + \frac{\partial^2\theta}{\partial y^2} + \frac{\partial^2\theta}{\partial z^2} = \frac{1}{\alpha}\frac{\partial\theta}{\partial t} \tag{1}$$

With an instantaneous point source located at the origin equation (1) is satisfied by

$$\theta = \frac{S}{8(\pi\alpha t)^{3/2}} e^{-[x^2 + y^2 + z^2]/4\alpha t} \tag{2}$$

where θ is temperature rise

t is time after initiation of heat generation

α is thermal diffusivity

S is the strength of the source

The temperature field represented by θ is the temperature field in an infinite solid due to a quantity of heat instantaneously generated at $t = 0$ at a point located at the origin.

This can be extended to an instantaneous line source along the z axis by considering a distribution of point sources along a line so that θ is given by

$$\theta = \frac{S}{8(\pi\alpha t)^{3/2}} \int_{-\infty}^{\infty} e^{-[x^2 + y^2 + z^2]/4\alpha t}\, dz \tag{3}$$

Integration of equation (3) gives

$$\theta = \frac{S}{4\pi\alpha t} e^{- [x^2 + y^2]/4\alpha t} \tag{4}$$

For a continuous line source with constant strength s per unit length, the temperature distribution as a function of time becomes

$$\theta = \frac{s}{4\pi\alpha} \int_0^t e^{-r^2/4\alpha t} \frac{dt}{t} \tag{5}$$

where $r^2 = x^2 + y^2$

The equation for θ can also be written as

$$\theta = \frac{s}{4\pi\alpha} \int_{\frac{r^2}{4\alpha t}}^{\infty} e^{-u} \frac{du}{u} = - \frac{s}{4\pi\alpha} E_i \left(- \frac{r^2}{4\alpha t} \right) \tag{6}$$

where E_i is the exponential integral

$$- E_i (-v) = \int_v^{\infty} \frac{e^{-u}}{u} du$$

For small values of v

$$E_i(-v) = \gamma + \ln v - v + \frac{1}{4} v^2 + 0v^3 \tag{7}$$

where γ is a constant.

When $\frac{r^2}{4\alpha t}$ is very small the equation for θ becomes:

$$\theta = \frac{s}{4\pi\alpha} \ln \frac{4\alpha t}{r^2} - \frac{\gamma s}{4\pi\alpha} \qquad (8)$$

The use of the line source method for measuring thermal conductivity is explained in detail by Van der Held and Van Drunen(2) and the following discussion is extracted from reference (2).

The equation for θ can be recast into a more convenient form by noting that

$$s = \frac{q}{\rho c} \text{ and } \alpha = \frac{K}{\rho c}$$

where q is heat generated per unit length

ρ is density

K is thermal conductivity

c is specific heat

Substitution for q and α into equation (8) gives

$$\theta = \frac{q}{4\pi K} \left[\ln \frac{4\alpha t}{r^2} - \gamma \right] \qquad (9)$$

Actually γ is not a general constant and the equation includes α, however both of these difficulties can be avoided by taking the difference in temperature at two times to get

$$\theta_2 - \theta_1 = \frac{q}{4\pi K} \ln \frac{t_2}{t_1} \qquad (10)$$

The errors which will always be included in the application of the method are summarized as follows:

1. Effect of length - There will be heat losses at the ends on account of the finite length involved; however, the loss has been shown to be negligible if the temperature is measured at the center of the sample and the sample length is large compared to the heater wire diameter.(4)
2. Finite wire size - Since the wire must have a finite size an additional heat production is required to heat the wire.
3. Starting time - It is difficult to determine the precise time that the heating starts and consequently an error in time measurement is inherent.
4. Inherent mathematical error - A mathematical error arises from the truncation of the series for evaluating E_i (v).

Van der Held and Van Drunen have shown quite conclusively that the errors 2, 3 and 4 can be accounted for by including a time correction t_o so that

$$\theta_2 - \theta_1 = \frac{q}{4\pi K} \ln \frac{t_2 + t_o}{t_1 + t_o} \tag{11}$$

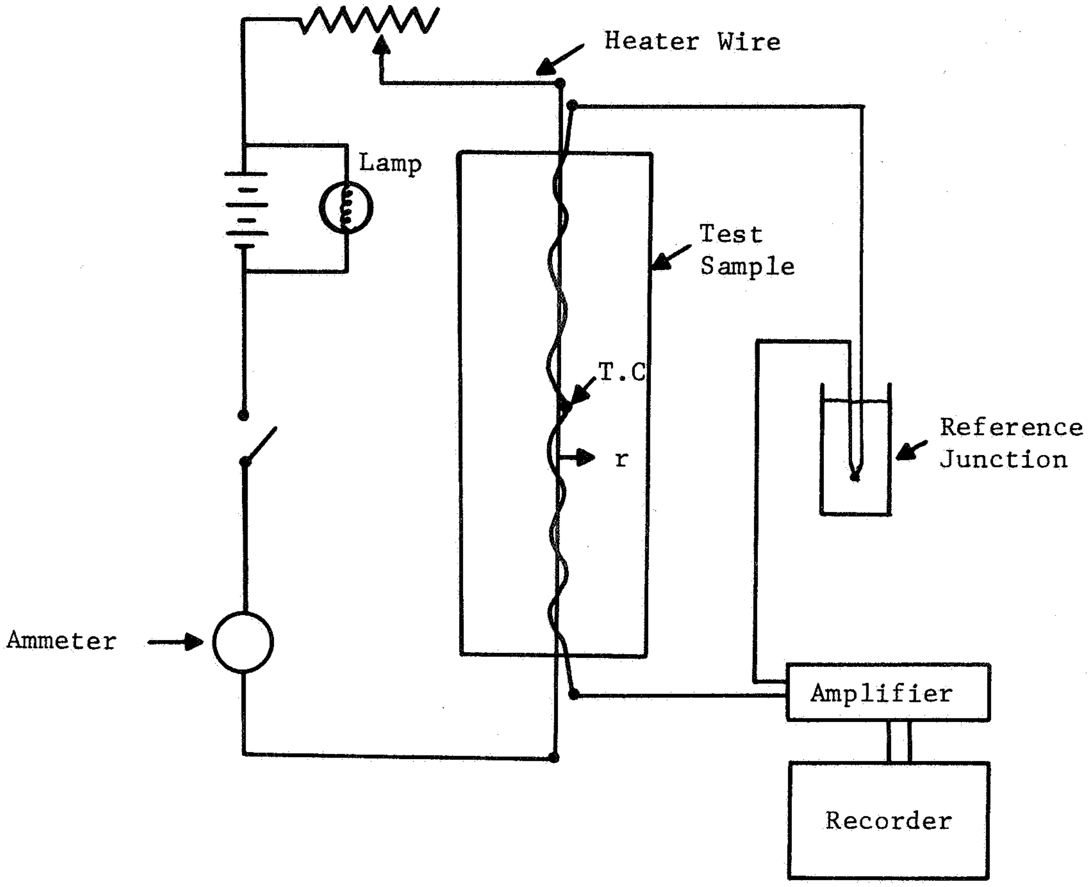
One means of evaluating t_o is by considering

$$\frac{\partial t}{\partial \theta} = \frac{4\pi K}{q} (t + t_o) \tag{12}$$

If $\partial t/\partial\theta$ is plotted vs. time a straight line with slope = $4\pi K/q$ results. The intercept for $\partial t/\partial\theta = 0$ is $-t_o$. Although the conductivity K could be obtained from the slope of this curve, it is better to determine K from equation (11) because of the error resulting from differentiation of the experimental curve.

A schematic diagram of the type of experimental equipment required is shown in figure 1.

A typical set of results is illustrated in figure 2.



Heater Wire	30 gauge manganin
Thermocouple	30 gauge copper-constantan
Heater Wire Current	200 to 300 milliamperes
Temperature Rise	5 degrees Fahrenheit maximum
Time	0 - 30 seconds

Figure 1 Typical Experimental Arrangement

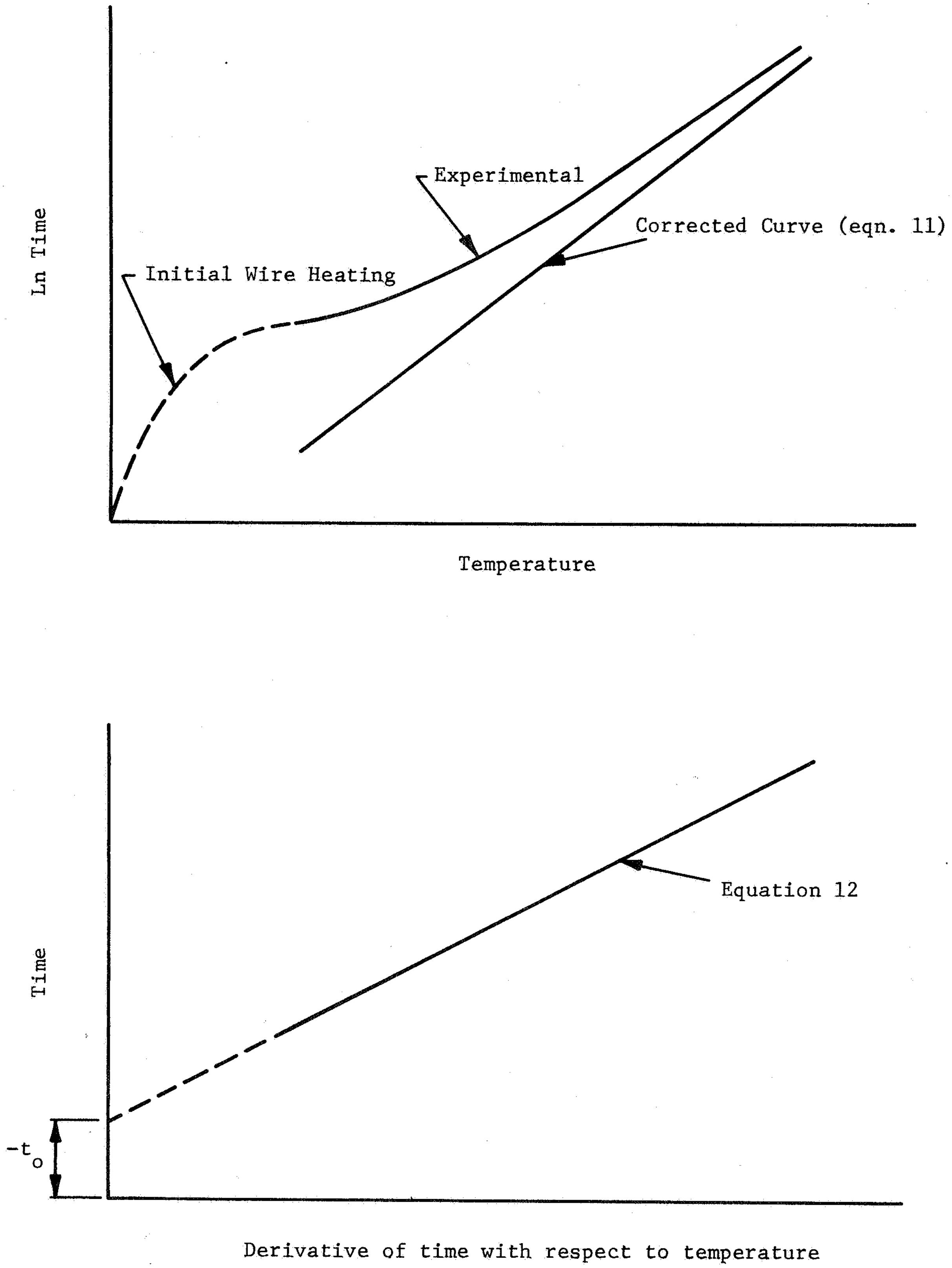


Figure 2 Line Source Data Reduction

Experimental Apparatus

Figure 1 illustrates the basic system required for use with the line source method. The only essential difference between the system actually used and the one shown in figure 1 is that the actual system was designed so that six separate samples could be tested by simply switching the recording instruments and battery to each sample in succession (see figure 3).

The system was designed so that six samples could be tested simultaneously--three samples in a vacuum chamber and three samples in a constant humidity chamber. The test apparatus is shown in figure 4. All of the instrumentation used is on the table between the vacuum system and the humidity chamber. The recording potentiometer on the extreme right was not used in the experiments. Figures 5 and 6 show the brackets used for mounting the samples in the humidity and vacuum chambers. The entire test apparatus was installed in an air-conditioned room where the temperature was maintained at 70±2F.

The instruments used (see figures 3 and 4 for location in the system) were as follows:

1. Recorder - Minneapolis-Honeywell recording potentiometer Model Y 153 X 17 (VA)-X-30DN6, variable span to 1.0 mv. full scale and 8 inches/min. chart speed
2. Ammeter - Westinghouse Type PX-161 DC Milliammeter
3. Potentiometer - Rubicon Model 2745
4. Amplifier - Hewlett Packard Model 2470A Data Amplifier
5. Vacuum gauge - NRC type 507 ionization gauge with NRC type 710R Vacuum Gauge Control

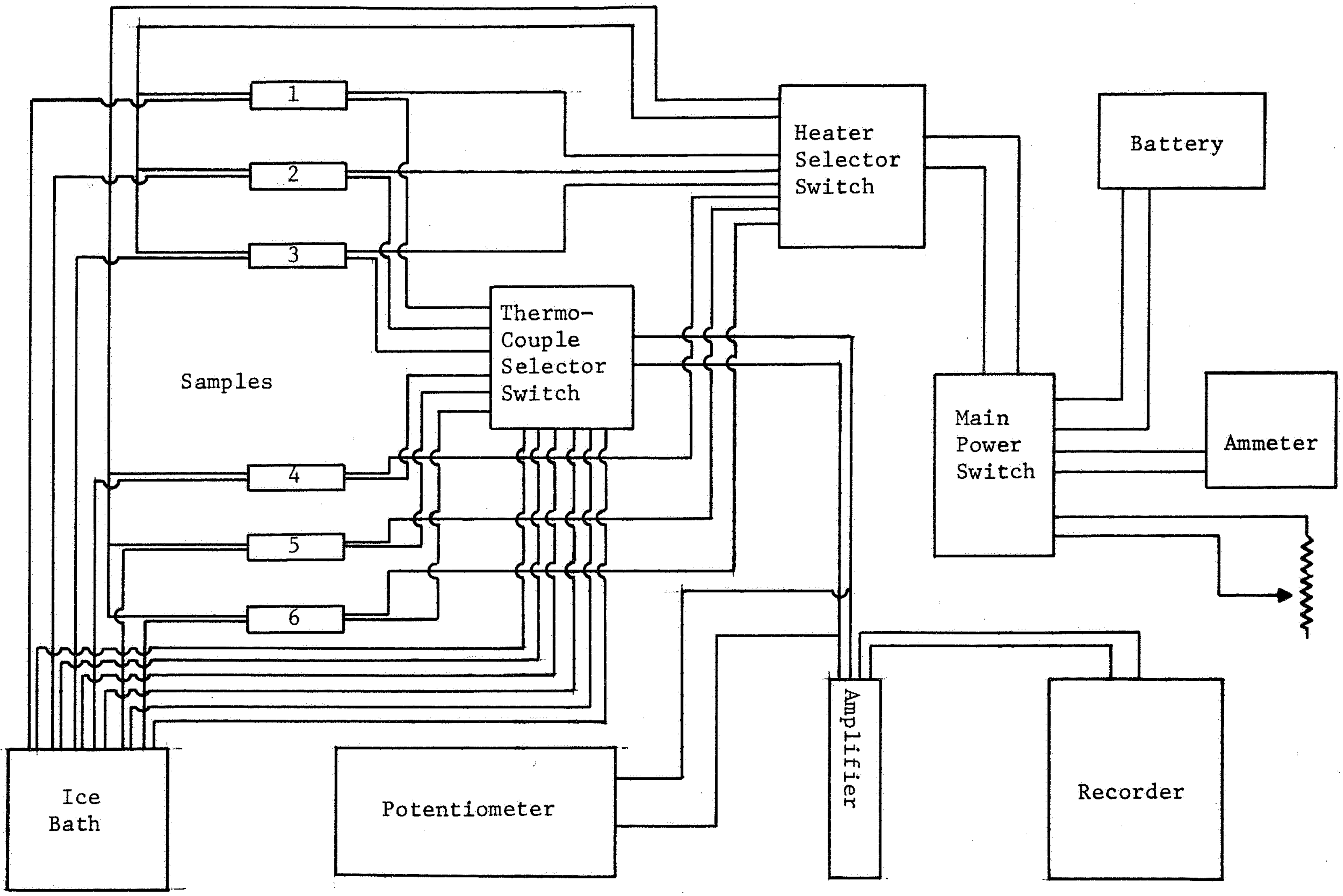


Figure 3 Wiring Diagram for Line Source Method

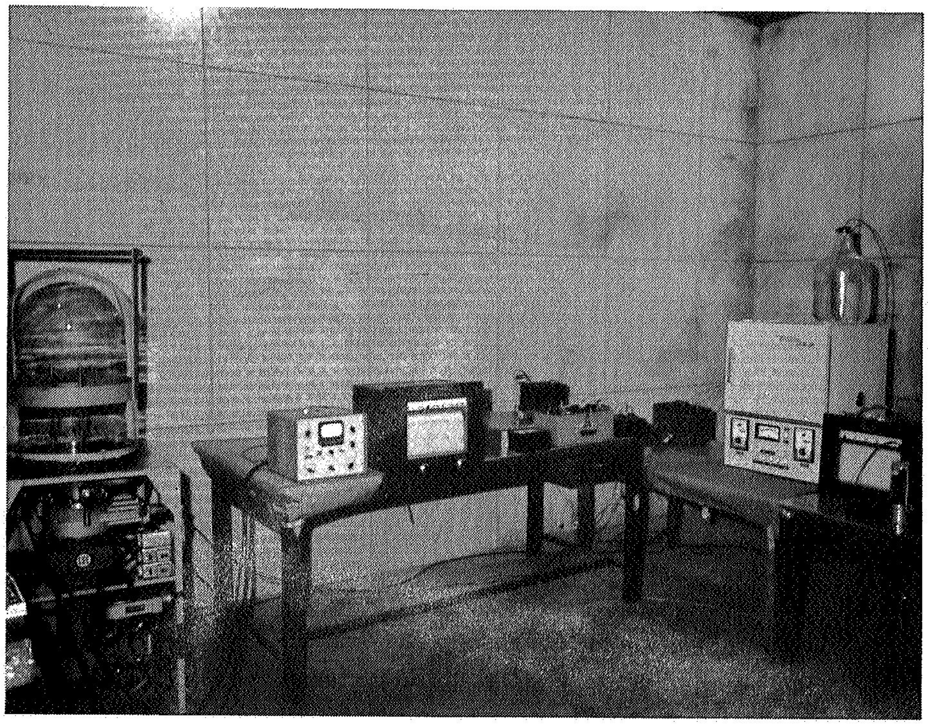


Figure 4 Test Apparatus

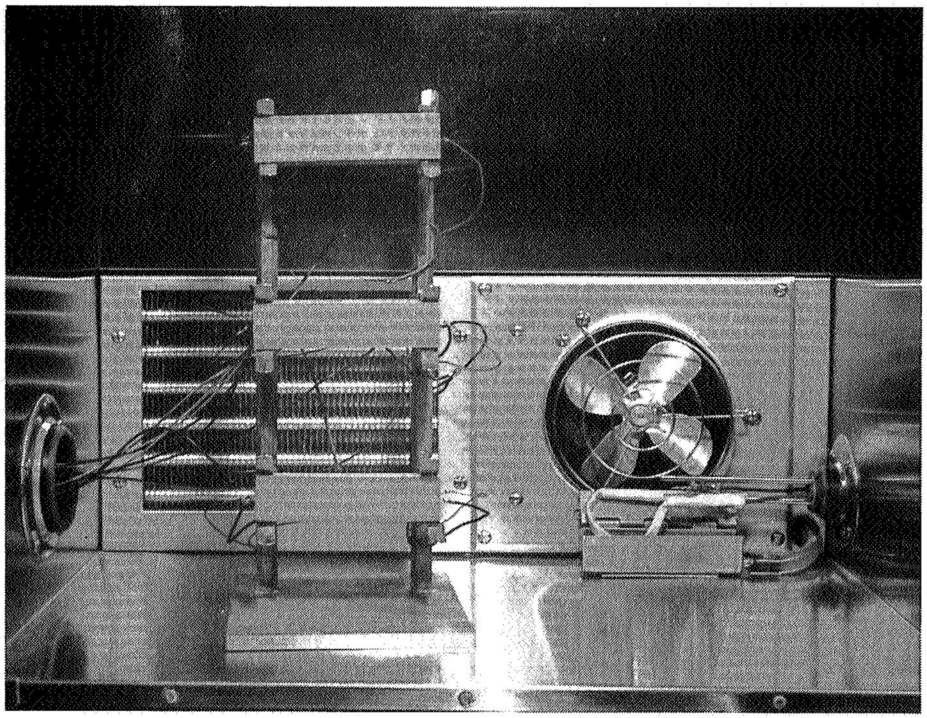


Figure 5 Sample Mounting in the Humidity Chamber

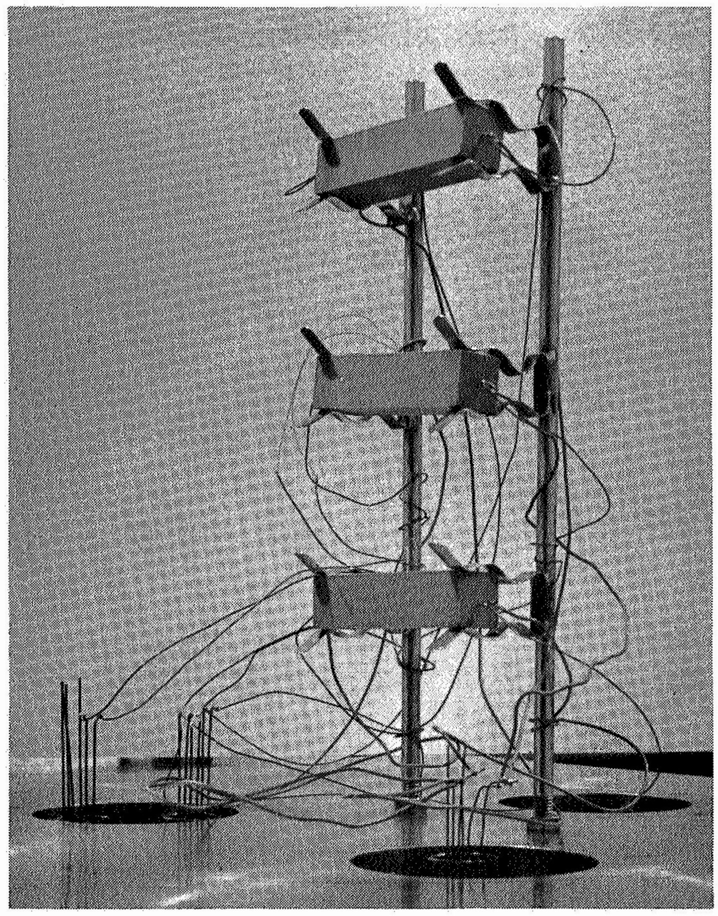


Figure 6 Sample Mounting in the Vacuum Chamber

The vacuum chamber used was an 18-inch bell jar arrangement coupled to a CEC 4-inch diffusion pump and a Welch roughing pump. The vacuum system included a water-cooled chevron baffle and the ultimate vacuum capability of the system was 10^{-7} torr with no load.

The constant humidity chamber used was a Tenney Engineering, Inc. Model TH-Jr. The unit is capable of controlling dry bulb temperature within ± 1/2°F and relative humidity within ± 1% at 90% or higher and ± 5% at lower humidities.

A simple comparator was also constructed so that the conductivity of a sample could be obtained by comparing the temperature drop across the sample with the temperature drop across a reference sample with a known conductivity. A schematic of the apparatus is shown in figure 7. The temperatures in the comparator were monitored with the same potentiometer that was used for the other experiments. The comparator was designed to utilize round samples with a 4-inch diameter. Various thicknesses could be used by varying the size of the spacers. Typical samples used were approximately one inch thick. The thermocouples were mounted in 1/8-inch thick copper plates which were placed as shown in figure 7.

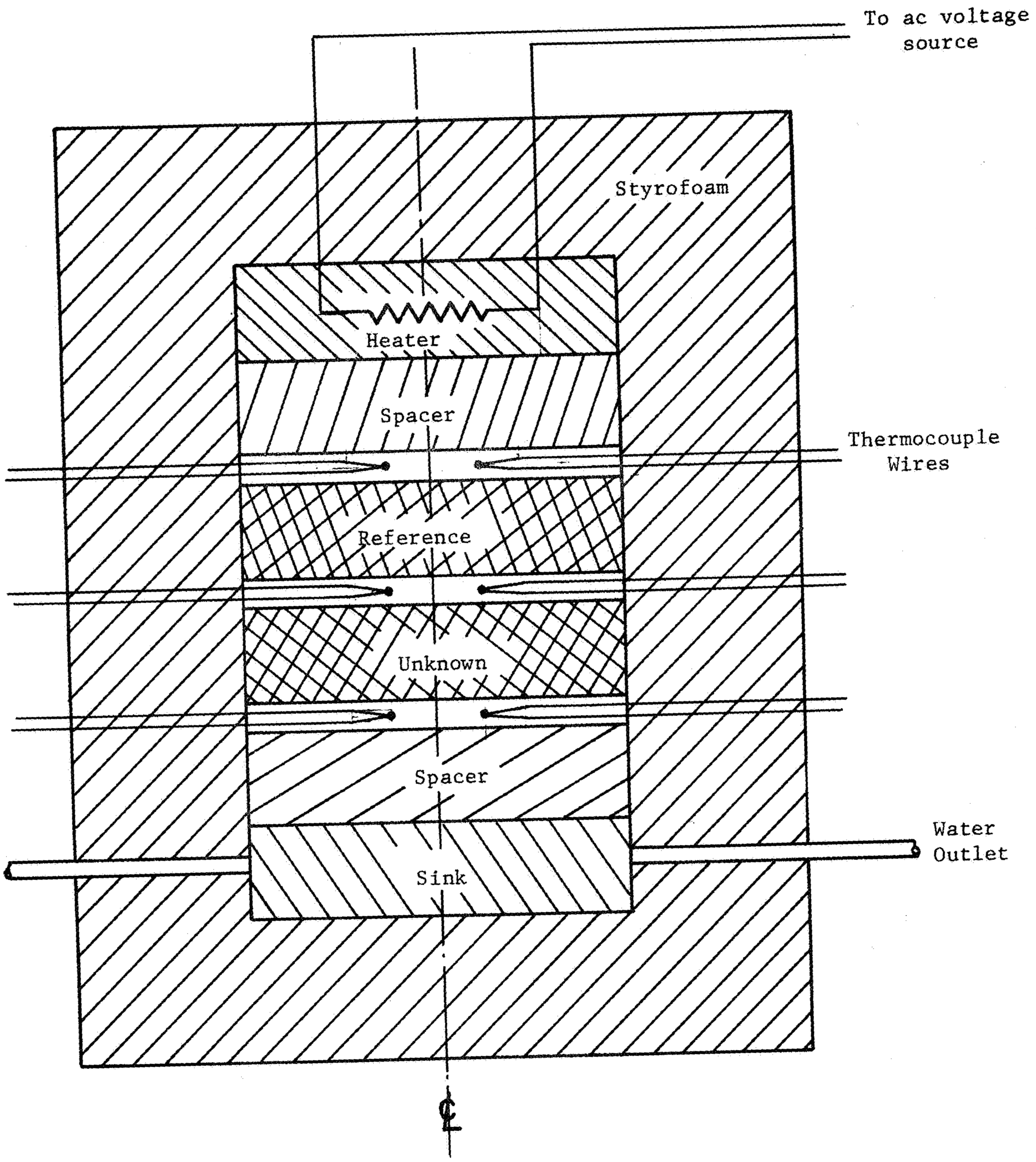


Figure 7 Comparator

Sample Preparation

The heat shield material used in these tests was NASA E4A1 with a composition by weight as follows: 73.0% General Electric RTV 602 elastomer, 11.0% Emerson and Cumming, Inc. silicon eccospheres, 10.0% Union Carbide Plastic Co. phenolic microballons, 4.0% Johns Manville Fiberglass, Inc. microquartz fibers, 2.0% General Electric SF-69 dimethyl silicone oil, and 0.5% General Electric SRC-04 RTV 602 curing catalyst (based on weight of elastomer).

The samples tested were one-inch square cylinders four inches long. Since the sample shape used with the line source method is not important the square cylinder was chosen for its simplicity.

The sample size is also not important as long as it is large enough so that the temperature propagation does not reach the surface during the testing period. The initial choice of sample size was based upon the recommendations of Underwood and McTaggart(4), who tested a material with approximately the same properties as the material tested in this investigation. The experimental results indicate that the samples were large enough.

The samples were cut from a large block of material with hand tools. To avoid contamination of the samples the following precautions were observed:

1. The material was kept in moisture-proof polyethlene bags during storage and handling, except during the actual cutting or experimentation.

2. When the material was handled clear plastic gloves were always worn.

3. All cutting tools, jigs and wires used were cleaned with ethyl alcohol immediately before use.

The difficult part of the sample preparation was the insertion of the heater wire and the thermocouple into the samples. The following procedure was used. (See figure 1 for a schematic diagram of the test sample.)

A hypodermic needle (0.028 in. O.D.) was forced through the sample using the jig shown in figure 8. The hypodermic needle was fed through the lathe chuck in small increments to prevent bending and the penetration was accomplished by moving the compound support to which the jig was attached. The lathe chuck was rotated slightly by hand to facilitate penetration of the sample. During the penetration process a smaller diameter hypodermic tubing was inserted into the tubing held in the chuck to provide increased stiffness.

After the tubing had penetrated the entire length of the sample the inner tube was removed and a length of heater wire was inserted into the remaining tube. One end of the thermocouple wire was bent into a hook which could be inserted into a slot cut near the end of the hypodermic tubing. The thermocouple wire was then wrapped around the heater wire in a helical pattern. The hypodermic tubing was then extracted from the sample and the heater wire-thermocouple combination was pulled through the sample following the tubing until the thermocouple junction was at the center of the sample.

The thermocouple and heater wires were clipped approximately one-fourth of an inch from the ends of the sample and larger diameter lead wires were soldered to the ends.

The whole process must be done carefully and slowly to avoid breaking the wires, and after a few trials no particular difficulty was encountered.

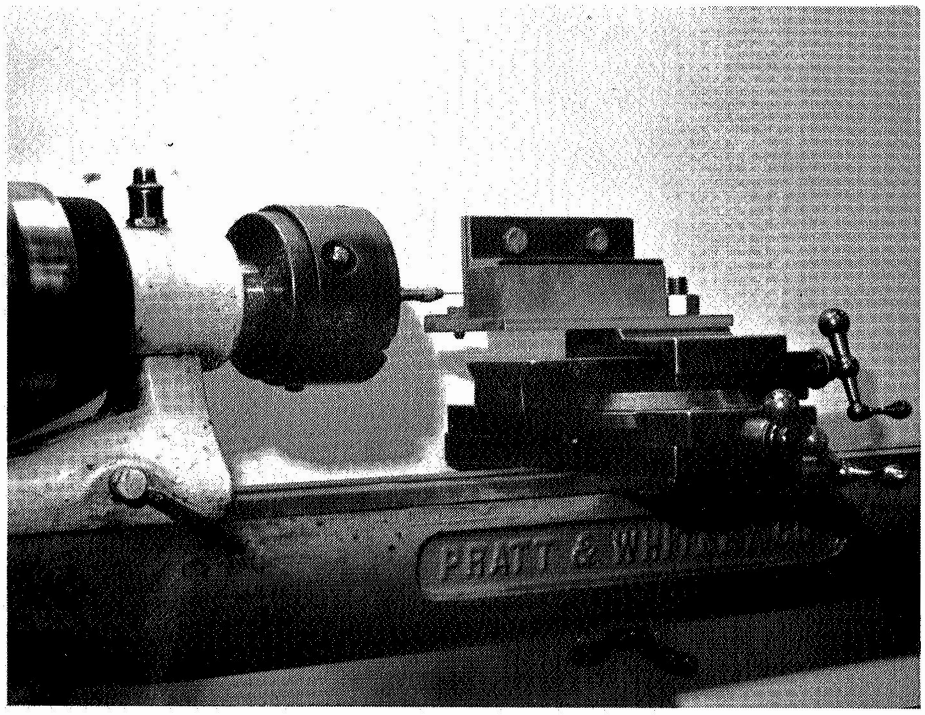


Figure 8 Jig for Sample Preparation

The hypodermic tubing used was only slightly larger than the combined heater and thermocouple wires and the material was sufficiently elastic to provide a tight fit for the wires.

Test Procedure

One of the advantages of the line source method is that the experimental technique required is quite simple.

After the entire system had been prepared and all of the samples were installed, the electrical circuits were checked and the system was allowed to stand for 24 hours prior to beginning the actual test to insure that all the samples were in thermal equilibrium.

Thermocouples were inserted near the surface in two of the samples to check for thermal equilibrium. These thermocouples were inserted in sample number one which was in the vacuum chamber and in sample number four which was in the constant humidity chamber. Prior to each run the voltages were measured for the thermocouples in all of the samples so that the surface temperatures could be compared with the interior temperatures. The results are tabulated in Appendix D. There was no significant difference between the surface and interior temperatures.

On the first day of the test, data were taken for all the samples at ambient conditions and then the vacuum system and the humidity chamber were put into operation. Subsequent testing was done at 24-hour intervals. After nine days both the vacuum system and the humidity chamber were turned off and all six samples were exposed to the atmosphere. The testing was continued for three more days at ambient conditions.

Prior to each test, the calibration of the amplifier-recorder combination was checked with the Rubicon potentiometer. With the recorder constantly in operation the individual tests were started and stopped by simply turning the heater current on and off.

The actual test time for each sample was limited to less than forty seconds to prevent unnecessary heating of the samples in the vacuum chamber. Dissipation of the energy added to the samples during testing requires a long time because of the vacuum.

Results

A typical set of experimental data is shown in figure 9. The data are plotted for one sample for the initial condition, after three days in vacuum, and for the last day of the test which corresponds to three days at ambient conditions after vacuum exposure.

Consider the data for the first and last days of the test. The temperature rise is directly proportional to the power supplied to the wire so that if the temperature rise were adjusted for difference in the power levels the curves would coincide (for $t > 5$, or after the starting period). It is immediately apparent that the conductivity before and after vacuum exposure is at least approximately the same.

Since the thermal conductivity is inversely proportional to the slope of the curves for constant heat addition, a change due to vacuum exposure can be observed by comparing the initial data with the results after three days in the vacuum chamber. For these two conditions the difference in heater currents is small enough to be neglected and a direct comparison is possible.

While variations in thermal conductivity can be observed by studying the experimental data the actual values for the conductivity cannot be calculated until the correction factor in equation (11) has been determined.

The correction factors were obtained in the following manner:

1. The thermal conductivity was measured by use of the comparator described in the section on apparatus.
2. It was assumed that the initial conductivity was the same for each sample and equal to the value obtained with the comparator.

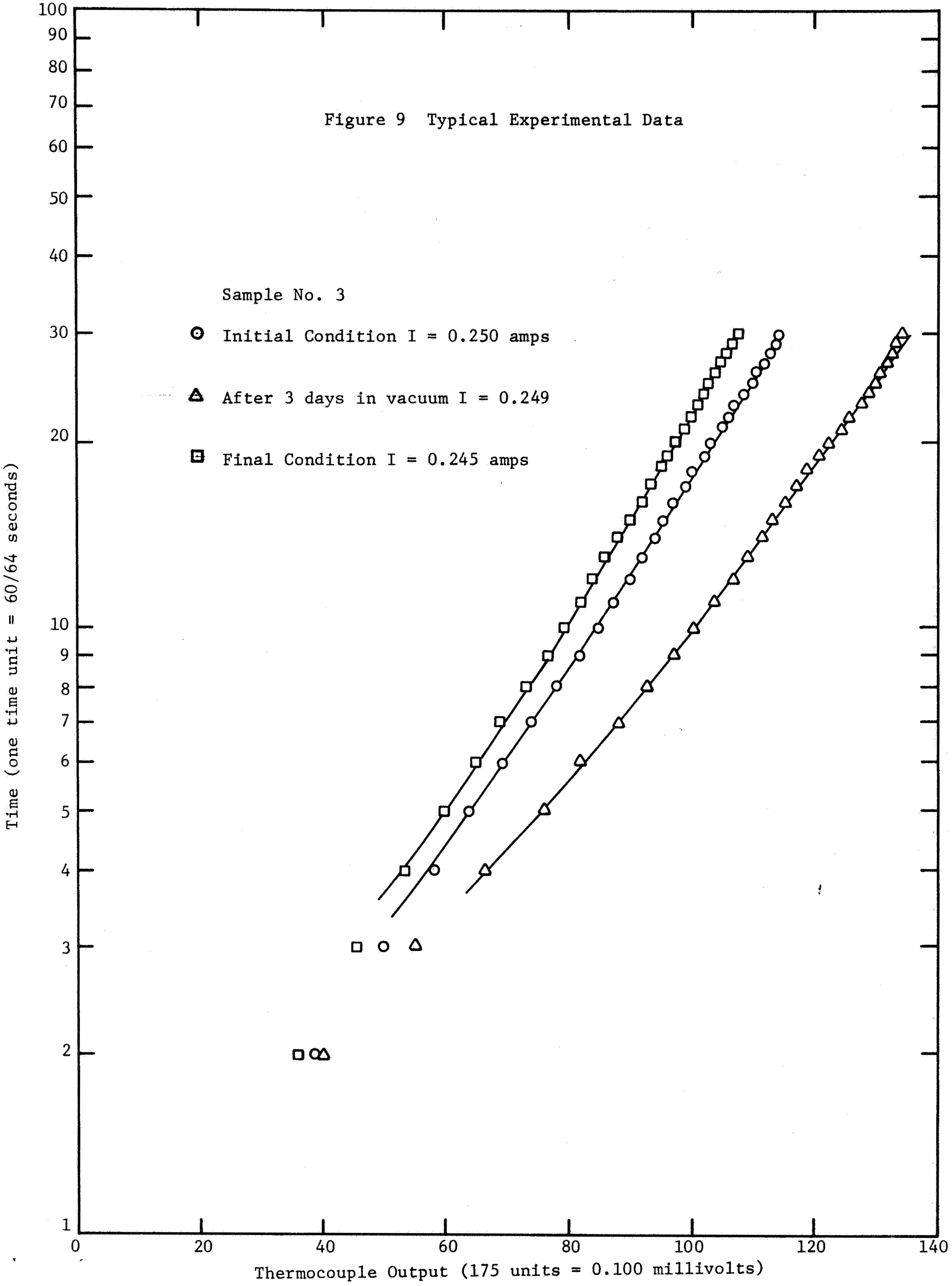


Figure 9 Typical Experimental Data

3. Equation (11) was solved to obtain the correction factor for each sample.
4. It was assumed that the correction for each sample remained constant during the entire test.

A detailed discussion and justification for the procedure used in determining the correction factors will be given in the section on Data Reduction.

The variation in thermal conductivity with time is given for each of the samples in figures 10 and 11. The average values of conductivity for both the vacuum samples and the control samples are shown in figure 12 together with the pressure variation. Some of the scattering of data for sample number 2 is due to a damaged electrical connection caused by the stress on the wire during sample preparation which was not apparent when the testing began. The results for sample number 2 are not included in the average values shown in figure 12. The thermal conductivity values are also listed in table 1 for convenience.

As indicated in figure 12 the thermal conductivity was reduced by approximately fifteen per cent due to the vacuum exposure and then returned to approximately the initial value upon subsequent reexposure to the atmosphere.

The relative humidity in the humidity chamber was held at 55 per cent. The humidity before the test was 30 per cent and for the three final days it varied between 25 and 34 per cent. The conductivity appears to be independent of relative humidity in the range of 25 to 55 per cent.

Tabulated experimental data are included in Appendix C.

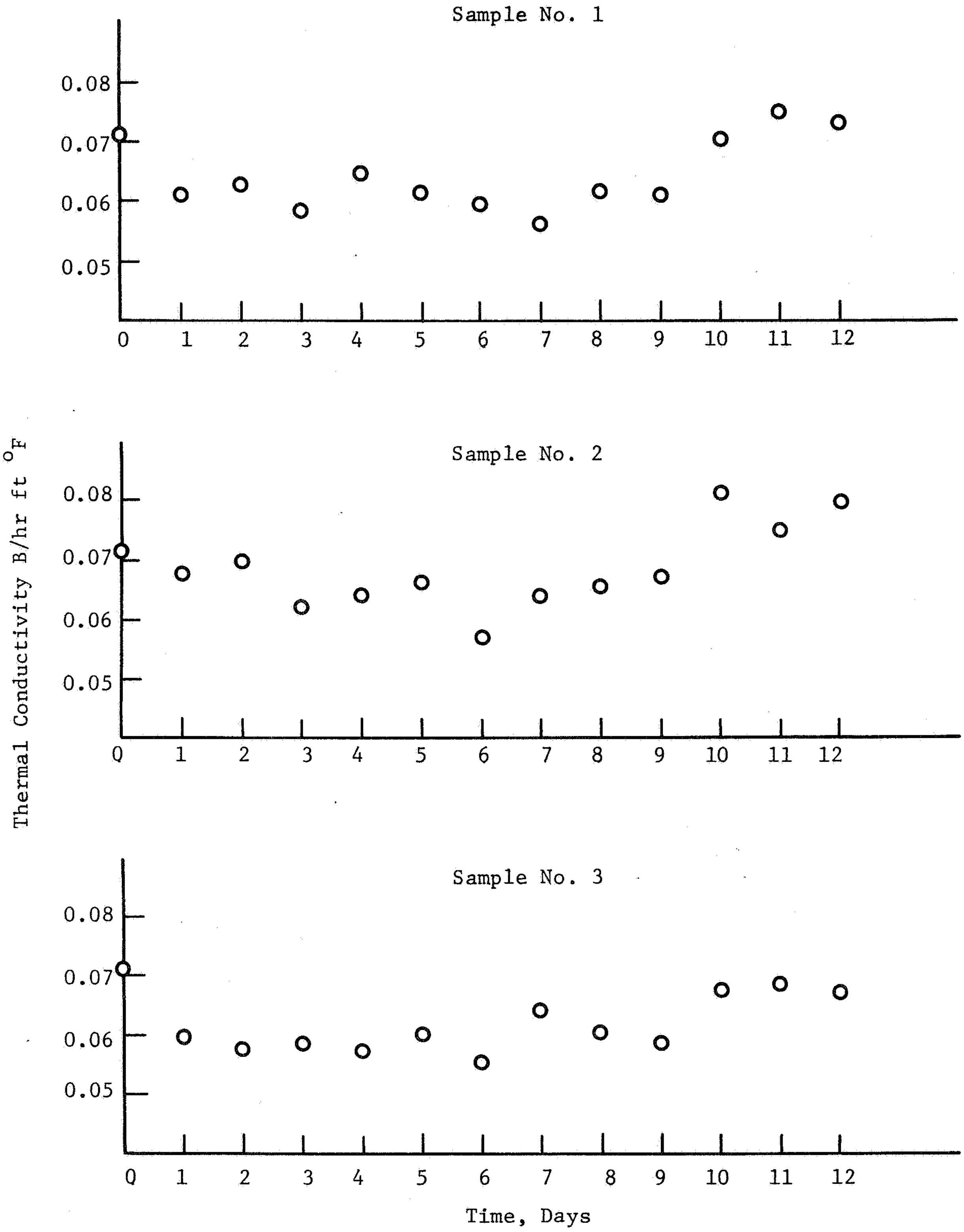


Figure 10 Thermal Conductivity Variation in the Vacuum Chamber

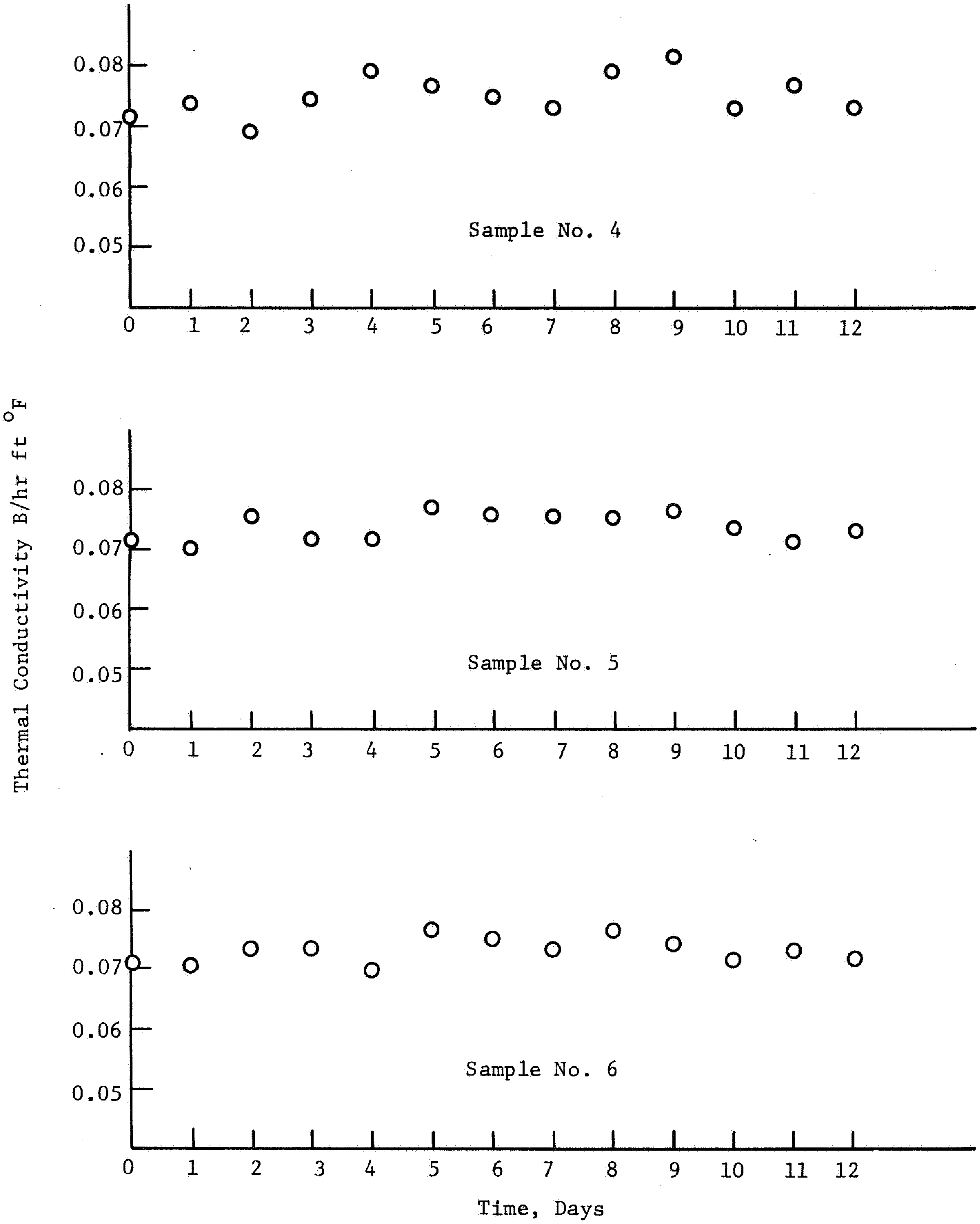


Figure 11 Thermal Conductivity Variation in the Humidity Chamber

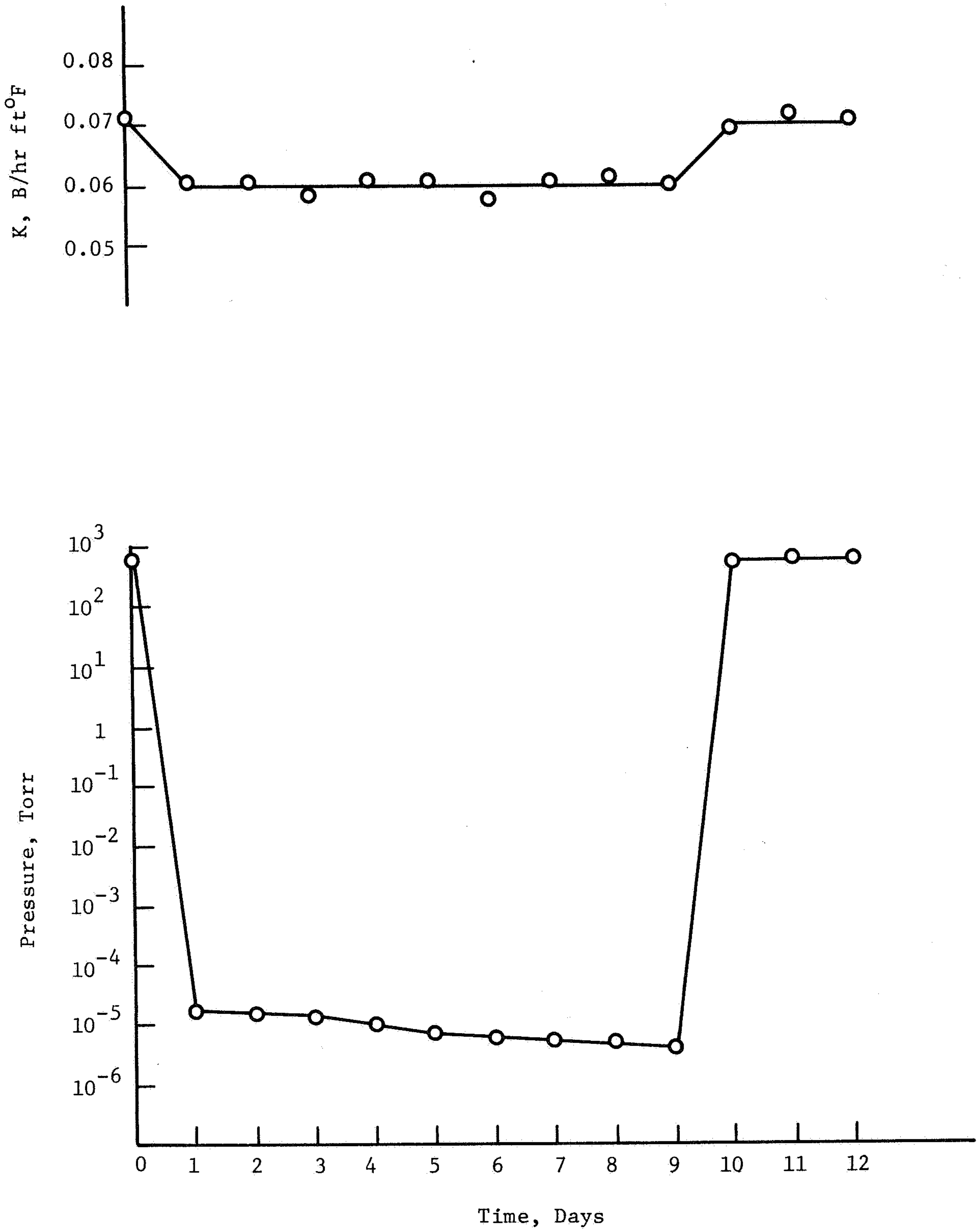


Figure 12 Average Thermal Conductivity and Vacuum Chamber Pressure

Table 1

Thermal Conductivity Values

All values are in (B/hr ft F) x 10^2

	Sample Number							
Run No.	1	2	3	Average (1 and 3)	4	5	6	Average (4, 5, and 6)
1	7.10	7.10	7.10	7.10	7.10	7.10	7.10	7.10
2	6.09	6.76	5.95	6.02	7.36	7.00	7.04	7.13
3	6.27	6.99	5.78	6.02	6.90	7.54	7.38	7.04
4	5.85	6.21	5.83	5.84	7.42	7.15	7.38	7.32
5	6.48	6.40	5.71	6.09	7.90	7.15	6.99	7.35
6	6.15	6.62	6.00	6.07	7.65	7.71	7.69	7.68
7	5.95	5.69	5.53	5.74	7.48	7.59	7.50	7.52
8	5.63	6.40	6.42	6.02	7.30	7.53	7.32	7.38
9	6.18	6.56	6.03	6.10	7.90	7.53	7.69	7.71
10	6.10	6.74	5.88	5.99	8.18	7.65	7.44	7.76
11	7.03	8.11	6.78	6.90	7.30	7.36	7.15	7.27
12	7.48	7.50	6.88	7.18	7.65	7.14	7.32	7.37
13	7.30	7.98	6.73	7.01	7.30	7.30	7.20	7.27

Data Reduction and Error Analysis

Equation (11) is the governing equation for the line source method and it is repeated here for convenience.

$$\theta_2 - \theta_1 = \frac{q}{4\pi k} \ln \frac{t_2 + t_o}{t_1 + t_o} \qquad (11)$$

The difficult part of the data reduction is the determination of the correction factor t_o. Note that if the experimental data were plotted on a semi-logarithmic coordinate system the experimental data would follow a curved line as illustrated in figure 1. The correction factor t_o is a constant having a value such that if t_o were added to each value of t the resulting curve would be a straight line.

A simple method for solving the problem has been suggested by Underwood and McTaggart (4). They suggest that the problem can be solved graphically if the experimental data are plotted on semilog coordinate paper. If a straight edge is placed along the points and adjusted until it lies an equal increment of time (not distance) away from each point then the corrected curve has been determined. The actual value of t_o is not of particular interest since the value of the thermal conductivity can be determined from the slope of the curve. Although this method is simple and is actually easy to use, the results are not satisfactory. It is difficult to get consistent results and if two different persons analyze the same set of data the results can easily differ by twenty per cent.

A more precise method for determining t_o has been suggested by Van der Held and Van Drunen (2) and involves the use of equation (12) as explained previously. A plot of time vs the derivative should produce a straight line and then t_o could be found easily as the intercept when the derivative is zero. This technique presents some difficulties because it requires precise determination of the derivative of time with respect to temperature.

In the present investigation it was found that equation (12) could be used over only a limited time interval (usually from about 6 to 18 seconds), and even then considerable scatter of the data was sometimes encountered. The difficulty is due to the fact that the correction factor is directly proportional to the derivative of time with respect to temperature. No matter what scheme is used to evaluate the correction factor, and even if the scheme does not involve a direct evaluation of the derivative, the fundamental dependence upon the derivative is still there.

The particular difficulty encountered in this investigation can be traced to a lack of resolution in the data recording system. The thermocouple output was amplified and then recorded using a recording potentiometer. The amplifier and recorder combination was calibrated as a unit using the Rubicon potentiometer as a reference. The calibration was accurate to within 1 part in 175. (The chart paper used was divided into divisions of approximately one millimeter and it was convenient to relate 175 chart units to a voltage difference of 0.100 millivolts.) The chart could be read to ± 0.5 scale units and the total change in voltage for a typical test was from 100 to 130 scale units so that the overall accuracy was within one per cent. The chart speed was 8 in./min (controlled by a synchronous motor), and since a typical test time was 30 seconds, the overall accuracy was more than adequate. It was convenient to use

1/8-inch increments in reading the chart output so the basic time unit used in the data is 60/64 seconds. (Only time ratios are used, so the particular value of the time unit is not important and no inconvenience is introduced as a result of the fraction.) The only other measurement required was the current in the heater wire and this was measured with a laboratory quality milliammeter accurate to within ± 0.25 per cent.

The overall accuracy of the data was more than adequate; however the resolution was not satisfactory for a precise determination of the derivative of time with respect to temperature. When a test was first started the voltage climbed rapidly in a small interval of time and then, as a result of the logarithmic relationship between the variables, the voltage change was small over a long period of time. There was only a small time interval (usually 6 to 18 seconds or less) during which the changes in time and temperature were of the same order (in terms of scale units). Thus there was only a short time period during which the derivative could be evaluated, and even then the precision was limited by the lack of resolution in the data.

To overcome some of these difficulties, considerable time was spent analyzing the data and the following finally emerged.

The data were read from the original recorder charts using 30 time increments of 60/64 seconds each and the data were then put on punched cards to facilitate computer operations. The computer was used to match a 4th order polynomial to the data for each individual run and the derivative of time with respect to temperature was calculated, also by computer, for each individual run as a function of time. For each sample the derivatives were plotted to evaluate the correction factor t_o. Figure 13 shows some of the data for sample number 3. The data shown are representative and some of the runs are omitted to make the results clear.

The range of data presented in figure 13 is restricted to the time interval in which the data can be represented by a straight line, since this is the only data that are useful in the evaluation of the correction factor. As noted previously the resolution of the data is inadequate to permit the evaluation of the derivative of time with respect to temperature outside a limited range.

It is obvious from the data presented in figure 13 that a precise value for the correction factor cannot be obtained from the graphical results. The assumption made previously that t_o is constant for a particular sample cannot be completely justified, however the data do indicate a trend toward a common correction factor so the assumption is reasonable.

On the basis of a constant correction factor for a given sample, the final data reduction was accomplished by calculating the correction value using equation (11) and a value of thermal conductivity determined by a comparison technique.

The comparison technique mentioned above was actually used initially for a different purpose. To provide a reference, a calibrated thermal conductivity reference specimen was obtained from the National Bureau of Standards. The reference specimen was gum rubber with a thermal conductivity of 0.089 B/hr ft ^{o}F which is in the range of conductivity expected for a material of the type being tested.

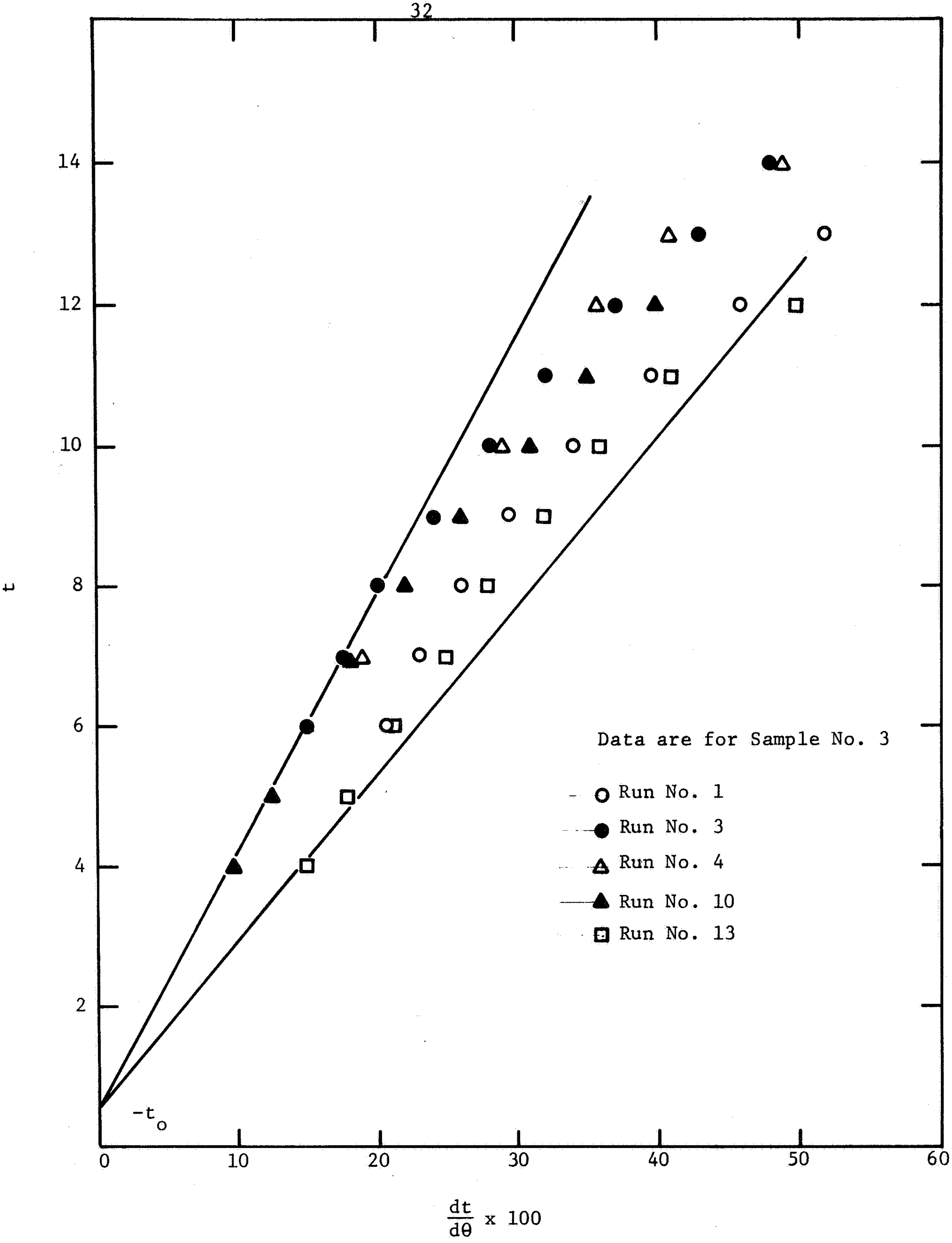


Figure 13 Determination of Correction Factor
(See Appendix A for scale factors for t and θ)

A simple comparison device was built, as shown in figure 7, into which a piece of the reference material could be inserted together with a piece of the test material. Assuming the same heat flow through the reference and unknown specimens the thermal conductivity can be deduced by a simple comparison of temperature differences. The device is admittedly crude, however it was intended for use only as a guide. When it was decided to use the data obtained with the comparator, a simple check was made to evaluate the accuracy of the technique. The unknown sample was replaced with another piece of the reference material and the temperature differences across the two reference samples were compared. The temperature difference across the piece which replaced the unknown was six per cent higher than the temperature across the reference sample. If the temperature differences were used to calculate the thermal conductivity by comparison there would be a six per cent error. The error can be attributed to heat transfer into the comparator from the surroundings, because the test sample was at a temperature below ambient.

Certainly the conductivity which was obtained with the comparator and used in the data reduction is not the true value, however it is a reasonable value and the deviation from the true value should be less than six per cent. The reason for assuming less than six per cent is that with the sample of the test material in the comparator the temperature inside the comparator was closer to ambient than with the NBS sample so that the heat leakage was less.

A further check of the method was attempted by measuring the conductivity of the reference material by use of the line source method.

A sample was cut from the reference material in the same shape and size as the test samples. The technique for sample preparation explained earlier would not work because the hypodermic tubing would not penetrate the gum rubber. The sample was finally cut in half with a saw and the heater and thermocouple wires were placed between the two pieces and the assembly was clamped with a parallel wood clamp. The results obtained with this technique indicated a value of thermal conductivity of 0.012 B/hr ft ^{o}F which was 26% greater than the value measured by the N.B.S. A significant part of this difference could be due to a variation in conductivity with direction in the reference material. It is also possible that the contact between the rubber and the heater wire is not complete.

Analysis

Since significant changes have been detected in the thermal conductivity of the test material due to vacuum storage, it would be helpful to have an explanation of the changes or at least some mathematical model which would help to predict the change.

The first approach to the question is to consider the work of Greenwood (1). Greenwood's work was concerned with the mechanical properties of a similar substance and a phenomenological model was employed to describe the behavior. The model used was concerned with the strength of the bond between the binder and the oxidizer in a solid propellant. Certainly the bond can be of utmost importance when considering mechanical properties, however when dealing with thermal properties the nature of the bond may assume a lesser importance. The ablation material being studied includes a large number of small hollow spheres, solid spheres, and fibers dispersed in a silicone rubber binder so that the physical contact between the binder and the filler materials will have some effect on the overall thermal conductivity. However, it seems more likely that the properties of the gas inside the hollow spheres will have a predominant effect. When the material is exposed to a vacuum, outgassing of the spheres would create voids which have negligible conductivity and this could provide an effect which overwhelms the effect of the bond between the binder and the filler materials.

Another approach to the explanation of conductivity changes is to consider the material as a porous solid. One approach has been considered by Harper and Sahrigi (5). They have proposed a model which includes a combination of continuous solid material and gas-filled cavities. This combination can be considered as providing both parallel and series paths for heat transfer. An analytical model has been developed from which it is possible to calculate the change in thermal conductivity due to changes in the properties of the gas included in the pores.

A prediction of conductivity was made based upon the analysis in reference 5 using a value for the conductivity of the binder obtained from reference 6. The results predict a conductivity of 0.076 B/hr ft °F when the porous material is filled with air and a conductivity of 0.133 B/hr ft °F with helium substituted for air.

An attempt was made to verify the theory experimentally and the results are shown in figure 14. The sample was placed in the vacuum chamber and after 24 hours (at a pressure of 10^{-5} torr) the chamber was filled with helium. After 24 hours with helium in the chamber the conductivity was measured again and then the sample was exposed to the atmosphere for 24 hours. The results are summarized in table 2. (See Appendix B for sample calculations.)

Table 2 Conductivity Measurements in Helium

Initial value of conductivity	0.071 B/hr ft °F
After 24 hours in vacuum	0.058 B/hr ft °F
After 24 hours in helium	0.086 B/hr ft °F
After 24 hours in atmosphere	0.068 B/hr ft °F

The value obtained in helium is significantly higher than the value in air, however it is considerably less than the predicted value. The theory includes empirical information which may not be exact, however it is also possible that the sample was not exposed to the vacuum and the helium for a sufficiently long period of time.

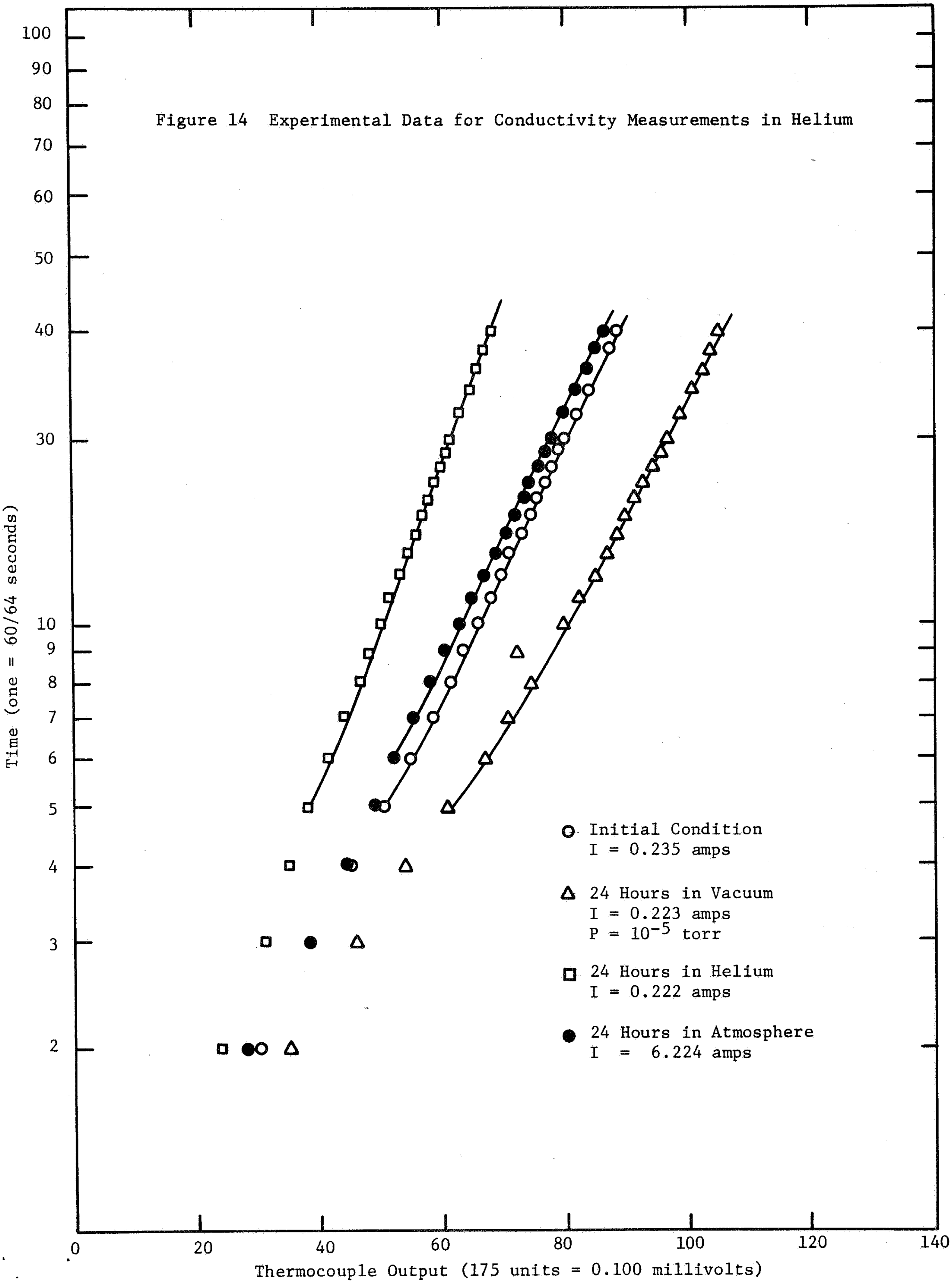


Figure 14 Experimental Data for Conductivity Measurements in Helium

Conclusions

The following conclusions can be drawn from the experimental investigation.

1. The thermal conductivity is reduced when the material is exposed to a vacuum. The magnitude of the reduction measured was approximately fifteen per cent using the data reduction technique given in this report. There is no doubt that the conductivity is significantly reduced, however the data reduction technique has not been verified.
2. In situ measurements are necessary because after reexposure to the atmosphere the thermal conductivity returns to its original value and there is no apparent effect due to vacuum exposure.
3. The thermal conductivity depends upon the properties of the environment to which the material is exposed. In some respects the material acts like a porous solid but the exact nature of its behavior in different environments has not been established.
4. The line source method is a promising technique for in situ measurements in different environments.

Recommendations

In the course of this project, a measurement system and a data reduction technique were developed in a short time. There are still many unknowns associated with the changes that were observed and the following suggestions are offered to aid in planning future studies.

1. The absolute accuracy of the technique used should be determined. One way to accomplish this would be to have the thermal conductivity of a sample measured by the National Bureau of Standards. If the conductivity of the same samples were subsequently measured by the line source method a conclusive evaluation of the technique could be made.
2. The line source technique should be further refined so that it can be used in a simple routine manner. The refinement needed includes a better data reduction technique and the determination of optimum sample size, heater current, and test duration.
3. An additional study of NASA E4A1 heat shield material should be undertaken to learn more about its behavior in a vacuum and other environments. For example, before a satisfactory analytical or phenomenological model can be devised to explain conductivity changes, it will be necessary to know whether the gases are dissolved in the binder or whether the gases are simply filling the cavities.

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Appendix A

Sample Calculations Using Equation 11

$$\theta_2 - \theta_1 = \frac{q}{4\pi K} \ln \frac{t_2 + t_o}{t_1 + t_o} \qquad (11)$$

$q = 3.413\ I^2 R$ B/hr ft

I = heater wire current in amperes

R = resistance of the heater wire = 2.8156 ohms/ft

$$\theta = \text{temperature } ^{o}\text{F} = \text{scale units} \times \frac{0.1 \text{ mv}}{175 \text{ scale units}} \times \frac{1^{o}\text{F}}{0.0225 \text{ mv}}$$

Substituting into equation (11) and solving for K gives,

$$K = \frac{30.11\ I^2}{\theta_2 - \theta_1} \ln \frac{t_2 + t_o}{t_1 + t_o} \quad \text{B/hr ft } ^{o}\text{F}$$

with I in amperes and θ in scale units

Time units used were 60/64 seconds, however no conversion factor is needed since only time ratios are used.

Appendix B

Calculation of the Thermal Conductivity of a Porous Solid by the Method Given in Reference 5

The governing equations are:

$$y = \alpha + \beta x + \frac{\zeta x}{(1 - \delta)x + \delta}$$

where $y = \frac{K_e}{K_s}$ and $x = \frac{K_g}{K_s}$

K_e = effective thermal conductivity

K_s = thermal conductivity of the solid

K_g = thermal conductivity of the gas in the pores

$K_e{}^*$ = effective thermal conductivity in high vacuum

$$\alpha = \frac{K_e{}^*}{K_s} = \psi(1 - \varepsilon)$$

$$\beta = 1 - \frac{\alpha^2}{2\alpha - (1 - \varepsilon)}$$

$$\zeta = \frac{\alpha(1 - \varepsilon) - \alpha^2}{2\alpha - (1 - \varepsilon)}$$

$$\delta = \frac{(1 - \varepsilon)}{\alpha} - 1$$

The equations were solved using the following values:

K_s = 0.220 B/hr ft ^{o}F from reference 6

K_e^* = 0.0616 B/hr ft °F average measured values

K_g(air) = 0.0148 B/hr ft °F from reference 7

K_g(helium) = 0.0847 B/hr ft °F from reference 7

ψ = 0.67 empirical constant from reference 5

Using the above constants,

$$y = 0.280 + 0.448x + \frac{0.272x}{0.507x + 0.493}$$

For air in the pores,

K_e = 0.076 B/hr ft °F

For helium in the pores,

K_e = 0.133 B/hr ft °F

Appendix C

Data For Thermal Conductivity

The data following are interpreted as follows:

Run No. 1 Initial data for all samples

Run No. 2-10 Vacuum Chamber and Humidity Chamber in Operation

Run No. 11-13 All samples exposed to atmosphere

Time one unit = 60/64 seconds

Thermocouple Output 175 units = 0.100 millivolts

```
                              SAMPLE NO 1



RUN NO        1        2        3        4        5        6        7
CURRENT     0.251    0.250    0.249    0.248    0.247    0.248    0.247


TIME                    THERMOCOUPLE OUTPUT

  1          17.0     15.0     22.0     21.0     26.0     23.0     20.0
  2          36.0     31.0     39.0     35.0     42.0     38.0     36.0
  3          48.0     46.0     50.5     49.5     53.0     50.0     49.5
  4          56.0     56.0     61.5     59.0     62.0     60.5     58.0
  5          63.0     65.0     70.0     67.5     69.5     71.0     66.0
  6          68.5     73.0     77.0     73.5     76.0     78.0     72.5
  7          72.5     79.5     82.5     78.5     82.0     84.5     78.5
  8          77.0     84.5     87.5     83.0     86.5     89.0     83.5
  9          80.5     89.0     92.0     87.0     90.5     93.5     88.0
 10          83.0     92.5     95.0     91.0     93.5     97.0     92.0
 11          86.0     95.5     98.0     95.0     96.5     99.5     95.0
 12          88.0     98.0    100.5     98.0     99.0    102.0     98.5
 13          90.5    100.0    103.0    101.0    101.0    105.0    101.0
 14          92.5    102.0    105.5    104.0    103.0    107.0    103.0
 15          94.5    104.5    107.5    106.5    105.0    109.0    105.0
 16          96.5    106.5    109.0    108.5    107.0    110.5    107.0
 17          98.0    108.0    111.0    110.5    109.0    112.5    108.5
 18         100.0    110.0    112.5    112.0    110.5    114.0    110.0
 19         102.0    112.0    114.5    113.0    112.0    116.0    111.5
 20         103.5    113.5    116.0    115.0    113.5    117.5    113.0
 21         105.0    115.0    117.5    116.5    115.0    119.0    114.5
 22         106.5    117.0    119.0    118.0    116.5    120.5    116.0
 23         107.5    118.0    120.0    119.0    117.5    122.0    117.0
 24         109.0    119.5    121.5    120.5    119.0    123.5    118.0
 25         110.0    121.0    123.0    122.0    120.0    124.5    118.5
 26         111.0    122.5    124.0    123.0    121.5    126.0    119.5
 27         112.0    123.5    125.0    124.0    122.5    127.0    120.5
 28         113.0    125.0    126.0    125.0    124.0    128.5    121.5
 29         114.0    126.0    127.0    126.5    125.0    129.5    122.0
 30         115.0    127.0    128.0    127.5    126.0    130.5    122.5
```

SAMPLE NO 1 (CONTINUED)

RUN NO	8	9	10	11	12	13
CURRENT	0.249	0.247	0.247	0.246	0.246	0.245
TIME	THERMOCOUPLE OUTPUT					
1	20.0	25.5	20.0	24.0	25.0	23.0
2	34.0	40.5	37.0	40.0	39.0	38.0
3	45.5	52.0	50.0	49.0	49.5	49.0
4	55.5	61.5	60.0	57.0	56.0	56.0
5	64.0	70.0	67.5	63.5	62.5	63.0
6	71.5	76.5	74.5	69.0	68.0	69.0
7	78.0	82.0	80.0	74.0	72.0	74.0
8	83.5	87.0	85.0	77.5	76.0	77.5
9	88.5	91.0	90.0	81.0	79.5	80.5
10	92.5	94.5	93.5	84.0	82.5	83.5
11	96.0	97.5	97.5	86.5	85.0	86.0
12	99.0	100.0	99.5	89.0	87.0	88.5
13	102.0	103.0	101.5	91.0	89.0	90.5
14	104.5	105.0	104.0	93.0	91.0	92.5
15	107.0	107.0	106.0	95.0	92.5	94.0
16	109.0	109.0	107.5	96.5	94.0	96.0
17	110.5	110.5	109.5	98.5	95.5	97.5
18	112.0	112.0	111.0	100.0	97.0	99.0
19	113.5	114.0	112.5	101.5	98.5	100.0
20	115.0	115.5	114.0	103.0	100.0	101.5
21	116.0	117.0	116.0	104.5	101.0	103.0
22	117.5	118.5	117.5	106.0	102.0	104.0
23	119.0	120.0	118.5	107.5	103.5	105.5
24	120.0	121.0	120.0	108.5	105.0	106.5
25	121.0	122.5	121.0	110.0	106.0	107.5
26	122.0	124.0	122.0	111.0	107.0	108.5
27	123.5	125.0	123.5	112.0	108.0	109.5
28	124.5	126.0	124.5	113.5	109.0	110.5
29	125.0	127.5	126.0	114.5	110.0	111.5
30	126.0	128.5	127.0	115.5	111.0	112.5

SAMPLE NO 2

RUN NO	1	2	3	4	5	6	7
CURRENT	0.250	0.249	0.248	0.248	0.247	0.248	0.246
TIME	THERMOCOUPLE OUTPUT						
1	23.0	15.0	20.5	19.0	20.0	22.0	16.0
2	39.0	30.5	36.5	35.0	36.0	37.5	30.0
3	50.5	46.0	50.0	47.0	47.0	49.0	42.0
4	59.0	56.0	60.0	57.5	56.0	58.0	54.0
5	64.0	64.5	67.5	66.0	64.0	67.0	61.5
6	70.0	72.0	74.0	72.5	71.0	72.0	68.5
7	76.0	77.0	80.0	78.0	77.0	77.0	74.0
8	80.0	81.0	84.5	83.5	82.0	81.0	79.5
9	83.5	84.5	88.0	88.0	86.0	84.5	84.5
10	86.5	87.5	91.5	91.5	90.0	88.5	89.0
11	89.0	90.0	94.5	95.0	93.0	92.0	93.0
12	92.0	93.0	97.0	97.5	96.0	95.0	96.5
13	94.0	96.0	99.0	100.0	98.5	98.0	99.0
14	96.0	98.0	101.0	102.5	100.5	100.0	101.5
15	98.0	100.5	103.0	104.5	102.5	102.5	103.5
16	99.5	103.0	104.5	106.5	104.0	104.0	105.5
17	101.0	105.0	106.0	108.5	106.0	106.0	107.5
18	103.0	106.5	107.5	110.0	107.5	107.0	109.0
19	104.0	108.0	109.0	111.5	109.0	108.5	110.5
20	106.0	109.5	110.0	113.0	110.0	110.0	112.0
21	107.0	111.0	111.5	114.5	112.0	111.0	113.5
22	108.5	112.5	113.0	116.0	113.0	112.0	114.5
23	110.0	114.0	114.0	117.0	114.0	113.5	116.0
24	111.0	115.0	115.0	118.5	115.5	115.0	117.0
25	112.0	116.5	116.0	119.5	117.0	116.0	118.0
26	113.0	117.5	117.5	121.0	118.0	117.0	119.0
27	114.5	118.5	118.0	122.0	119.0	118.0	120.0
28	115.5	119.5	119.0	123.0	120.0	119.0	121.0
29	116.5	120.5	120.0	124.5	121.0	120.0	122.0
30	117.5	121.5	121.0	125.5	122.0	121.0	123.0

```
                             SAMPLE NO 2 (CONTINUED)



RUN NO        8        9        10       11       12       13
CURRENT     0.247    0.247    0.247    0.246    0.246    0.246


TIME                       THERMOCOUPLE OUTPUT

  1          20.0     22.5     16.0     23.0     19.0     23.0
  2          36.0     37.5     31.0     38.0     35.0     36.0
  3          46.0     49.0     44.0     48.0     46.5     46.0
  4          54.0     59.0     55.0     54.5     53.0     54.0
  5          62.5     67.0     63.5     60.5     59.0     60.0
  6          69.0     74.0     70.5     66.0     64.5     65.0
  7          75.5     79.0     76.0     70.0     69.0     69.0
  8          80.5     84.5     81.0     73.5     73.0     73.0
  9          85.0     88.5     85.0     76.0     76.5     76.0
 10          88.5     92.5     89.0     79.0     79.5     78.5
 11          91.5     95.5     91.5     81.5     82.0     81.0
 12          94.0     98.0     94.0     83.5     84.5     83.0
 13          96.0    100.0     96.0     85.5     87.0     85.0
 14          98.0    102.5     98.0     87.5     89.0     87.0
 15         100.0    104.0    100.0     89.0     90.5     89.0
 16         102.0    106.0    101.5     90.5     92.0     90.5
 17         103.5    107.5    103.0     92.5     93.5     92.0
 18         105.0    109.0    104.5     94.0     95.0     93.5
 19         106.5    110.5    106.0     95.0     96.5     95.0
 20         108.0    112.0    107.5     96.5     97.5     96.0
 21         109.0    113.0    109.0     98.0     98.5     97.5
 22         110.5    114.5    110.0     99.0    100.0     99.0
 23         111.5    115.5    111.0    100.5    101.0    100.0
 24         113.0    116.5    112.0    101.5    102.0    101.0
 25         114.0    118.0    113.5    103.0    103.0    102.0
 26         115.0    119.0    114.5    104.0    104.0    103.0
 27         116.0    120.0    115.5    105.0    105.0    104.0
 28         117.0    121.0    116.5    106.0    106.0    105.0
 29         118.0    122.0    117.5    107.0    106.5    106.0
 30         119.0    123.0    118.5    108.0    107.0    107.0
```

```
                              SAMPLE NO 3



RUN NO        1        2        3        4        5        6        7
CURRENT     0.250    0.250    0.248    0.249    0.248    0.248    0.247


TIME                      THERMOCOUPLE OUTPUT

  1          21.0     25.0     29.0     21.5     26.0     25.0     21.0
  2          39.0     43.0     46.0     40.0     43.0     44.0     38.0
  3          50.0     57.5     59.5     55.0     56.0     57.0     53.0
  4          58.5     67.0     67.5     66.5     66.0     68.0     68.0
  5          64.0     75.0     75.5     76.0     75.0     76.0     71.5
  6          69.5     83.0     83.5     82.0     82.0     82.0     78.5
  7          74.0     88.5     90.0     88.0     89.0     89.0     85.0
  8          78.0     93.0     95.0     93.0     94.0     94.0     90.5
  9          82.0     97.5     99.5     97.0     98.5     98.0     95.0
 10          85.0    101.0    103.5    100.5    102.5    102.0     99.0
 11          87.5    104.5    107.0    104.0    106.0    104.5    102.5
 12          90.0    107.5    110.0    107.0    108.5    107.0    105.0
 13          92.0    110.0    112.0    109.5    111.0    109.0    107.5
 14          94.0    112.5    114.5    111.5    113.0    111.0    109.5
 15          95.5    114.5    116.5    113.5    114.5    113.0    111.5
 16          97.0    116.5    118.0    115.5    116.5    115.0    113.5
 17          99.0    118.5    119.5    117.5    118.0    116.5    115.0
 18         100.0    120.0    121.0    119.0    120.0    118.0    117.0
 19         102.0    122.0    122.5    121.0    121.5    119.5    118.5
 20         103.0    123.0    124.0    122.5    123.0    121.0    120.5
 21         104.5    124.5    125.5    124.5    124.5    122.5    121.5
 22         106.0    126.0    127.0    126.0    126.0    124.0    123.0
 23         107.0    127.0    128.0    127.5    127.0    125.0    124.0
 24         108.5    128.0    129.5    129.0    128.5    126.5    125.5
 25         110.0    129.0    130.5    130.0    129.5    128.0    126.5
 26         110.5    130.0    132.0    131.0    131.0    129.0    128.0
 27         112.0    131.5    133.0    132.0    132.0    130.0    129.0
 28         113.0    132.5    134.0    133.0    133.0    131.5    130.0
 29         114.0    133.5    135.0    133.5    134.0    132.0    130.5
 30         114.5    134.0    136.0    134.5    135.0    133.0    131.5
```

```
                        SAMPLE NO 3 (CONTINUED)



RUN NO        8        9       10       11       12       13
CURRENT    0.248    0.247    0.247    0.246    0.246    0.245

TIME                   THERMOCOUPLE OUTPUT

  1         23.5     22.0     25.0     25.0     26.0     22.0
  2         40.0     40.5     44.0     38.0     39.0     36.0
  3         54.0     55.5     57.0     48.5     50.0     45.5
  4         66.0     65.5     67.0     57.0     57.5     53.5
  5         74.5     75.0     75.5     62.5     63.0     60.0
  6         82.0     83.0     83.0     68.0     68.0     65.0
  7         87.5     88.5     89.0     72.0     72.0     69.0
  8         92.5     93.0     94.0     76.0     76.0     73.0
  9         96.5     97.5     98.0     78.5     78.5     76.5
 10        100.0    100.5    101.5     81.0     81.0     79.5
 11        102.5    104.0    104.5     83.0     83.0     82.0
 12        105.0    107.0    107.5     86.0     85.0     84.0
 13        107.0    109.5    109.5     88.0     87.0     86.0
 14        109.0    111.5    112.0     90.0     89.0     88.0
 15        111.0    113.5    114.0     92.0     90.5     90.0
 16        112.5    115.0    115.5     93.5     92.0     92.0
 17        114.0    116.5    117.5     95.5     94.0     93.5
 18        115.5    118.5    119.0     97.0     95.0     95.0
 19        117.0    120.0    121.0     99.0     96.5     96.0
 20        118.5    121.5    122.5    100.5     98.0     97.5
 21        119.5    123.0    124.0    102.0     99.5     99.0
 22        121.0    124.5    125.5    103.0    101.0    100.0
 23        122.0    126.0    127.0    104.5    102.0    101.0
 24        123.0    127.0    128.0    105.5    103.0    102.0
 25        124.0    128.0    129.5    106.5    104.0    103.0
 26        125.5    129.5    130.5    107.5    105.0    104.0
 27        126.5    130.5    132.0    108.5    106.0    105.0
 28        127.5    131.5    133.0    109.0    107.0    106.0
 29        128.5    132.5    134.0    110.0    108.0    107.0
 30        129.5    132.5    135.0    110.0    109.0    108.0
```

SAMPLE NO 4

RUN NO	1	2	3	4	5	6	7
CURRENT	0.254	0.253	0.253	0.252	0.252	0.252	0.251
TIME				THERMOCOUPLE OUTPUT			
1	22.5	24.5	23.0	22.5	19.5	21.0	19.5
2	38.0	38.0	39.5	39.5	35.0	37.0	38.5
3	48.0	49.0	50.0	49.5	47.0	49.5	48.5
4	57.0	57.0	57.5	58.0	55.5	56.0	57.0
5	64.0	63.0	64.0	64.0	62.5	61.5	63.0
6	69.5	69.5	69.5	69.0	69.0	67.0	68.5
7	75.0	73.5	74.5	73.5	73.0	71.0	73.0
8	80.0	77.0	79.0	77.5	76.5	75.0	77.0
9	83.0	80.0	83.0	80.5	79.0	78.0	80.0
10	85.5	83.0	85.5	83.5	81.5	81.0	83.0
11	88.0	85.0	88.0	86.0	84.0	83.5	85.5
12	90.5	87.0	90.5	88.0	86.0	86.0	88.0
13	92.0	90.0	93.0	90.5	88.5	88.0	90.0
14	94.0	92.0	94.5	92.5	90.0	90.0	91.5
15	96.0	94.0	96.5	94.0	91.5	91.5	93.5
16	98.0	95.5	98.0	96.0	93.0	93.0	95.0
17	99.0	97.5	100.0	97.5	95.0	94.5	96.5
18	101.0	99.5	101.5	99.0	96.5	95.5	98.0
19	102.5	101.0	103.0	100.5	98.0	97.0	99.0
20	104.0	102.5	104.5	101.5	99.5	98.5	100.5
21	105.0	104.0	106.0	103.0	101.0	100.0	101.5
22	106.5	106.0	107.0	104.5	102.0	101.0	103.0
23	107.5	107.5	108.5	105.5	103.5	102.0	104.0
24	109.0	108.5	109.5	107.0	104.5	103.0	105.0
25	110.0	110.0	111.0	108.0	106.0	104.0	106.0
26	111.0	111.0	112.0	109.0	107.0	105.0	107.0
27	112.0	112.0	113.0	110.5	108.0	106.0	108.0
28	113.0	113.0	114.0	111.5	109.0	107.0	109.0
29	114.0	114.0	115.0	112.5	110.0	108.0	110.0
30	115.0	115.0	116.0	113.5	111.0	109.0	110.5

```
                         SAMPLE NO 4 (CONTINUED)



RUN NO       8        9       10       11       12       13
CURRENT    0.252    0.252    0.252    0.250    0.250    0.250


TIME                  THERMOCOUPLE OUTPUT

  1         23.0     22.0     27.0     25.0     21.0     19.0
  2         37.0     39.0     40.0     40.0     37.0     34.5
  3         49.0     48.5     50.0     48.5     48.0     45.0
  4         56.5     56.0     57.0     56.5     56.0     54.0
  5         63.5     62.0     63.0     62.0     62.5     60.0
  6         69.0     68.0     68.5     68.0     68.0     65.5
  7         74.0     72.0     73.0     71.5     72.5     70.0
  8         79.0     75.5     76.0     75.5     76.0     74.0
  9         82.0     79.0     79.0     78.0     79.0     77.0
 10         85.0     81.0     81.5     81.0     82.0     79.5
 11         87.5     83.0     83.5     84.0     84.5     82.0
 12         89.5     85.5     85.5     86.5     87.0     85.0
 13         91.5     87.5     87.5     88.5     88.5     87.0
 14         93.0     89.5     89.0     91.0     90.5     89.0
 15         95.0     91.0     91.0     93.0     92.0     90.5
 16         96.5     92.5     92.5     94.5     93.5     92.0
 17         98.0     94.0     94.0     96.0     95.0     93.5
 18         99.0     95.5     95.5     97.5     96.5     95.0
 19        100.5     97.0     97.0     99.0     98.0     97.0
 20        102.0     98.5     98.0    100.5     99.0     98.0
 21        103.0    100.0     99.5    101.5    100.0     99.5
 22        104.5    101.0    101.0    103.0    101.0    101.0
 23        105.5    102.0    102.0    104.0    102.0    102.0
 24        106.5    103.0    103.0    105.0    103.0    103.0
 25        107.5    104.0    104.0    106.0    104.0    104.0
 26        108.5    105.5    105.0    107.0    105.0    105.0
 27        109.5    106.5    106.0    108.0    106.0    106.0
 28        110.5    107.5    107.0    109.0    107.0    107.5
 29        111.5    108.5    108.0    110.0    108.0    108.5
 30        112.0    109.0    109.0    111.0    108.5    109.5
```

```
                                 SAMPLE NO 5



RUN NO         1         2         3         4         5         6         7
CURRENT      0.253     0.253     0.253     0.252     0.252     0.251     0.250


TIME                        THERMOCOUPLE OUTPUT

  1           25.5      27.0      33.0      26.0      27.0      15.0      24.0
  2           44.0      46.0      49.5      44.0      44.0      44.0      45.0
  3           56.5      57.0      59.0      56.0      54.0      54.5      55.5
  4           65.0      66.0      67.0      64.5      63.5      62.5      64.5
  5           71.5      72.0      74.0      70.5      70.0      70.0      71.0
  6           77.0      77.0      79.5      76.0      76.0      74.5      76.0
  7           81.5      81.5      84.0      80.5      81.0      79.0      80.0
  8           86.0      85.5      88.0      85.0      84.5      83.0      83.5
  9           89.5      89.5      91.0      88.0      88.0      86.0      86.5
 10           92.5      93.0      94.0      91.0      90.5      89.0      89.5
 11           95.5      96.0      96.5      94.0      93.0      91.0      92.0
 12           97.5      98.5      98.5      96.5      95.5      93.0      94.0
 13          100.0     100.5     101.0      99.0      98.0      95.0      96.0
 14          102.0     102.5     103.0     101.0      99.5      97.0      98.0
 15          103.5     104.0     104.5     102.5     101.5      98.5     100.0
 16          105.5     106.0     106.0     104.5     103.5     100.0     101.5
 17          107.0     107.5     108.0     106.0     105.0     101.5     103.0
 18          108.5     109.0     109.5     107.5     107.0     103.0     104.5
 19          110.0     110.5     110.5     109.0     108.5     104.5     106.0
 20          111.5     112.0     112.0     110.0     110.0     106.0     107.5
 21          113.0     113.5     113.0     111.5     111.5     107.0     108.5
 22          114.0     115.0     114.5     112.5     113.0     108.0     110.0
 23          115.5     116.5     116.0     113.5     114.0     109.5     111.0
 24          116.5     117.5     117.0     115.0     115.0     110.5     112.0
 25          118.0     119.0     118.0     116.0     116.5     111.5     113.0
 26          119.0     120.0     119.5     117.0     117.5     112.5     114.0
 27          120.0     121.0     120.5     118.5     118.5     113.5     115.0
 28          121.0     122.0     121.5     119.5     119.5     114.5     116.0
 29          122.0     123.0     122.5     120.5     120.5     115.5     117.0
 30          123.0     124.0     123.5     121.5     121.5     116.0     118.0
```

```
                         SAMPLE NO 5 (CONTINUED)



RUN NO       8         9        10        11        12        13
CURRENT    0.251     0.252     0.251     0.250     0.250     0.249

TIME                     THERMOCOUPLE OUTPUT

  1         26.0      29.5      23.0      26.0      26.0      28.0
  2         42.5      45.0      40.0      43.5      43.0      44.5
  3         54.0      55.5      52.5      54.5      54.0      56.0
  4         62.0      63.0      62.0      64.0      61.0      64.0
  5         69.0      69.0      68.5      70.0      67.5      70.5
  6         74.5      75.0      75.0      75.5      73.5      76.0
  7         79.0      80.0      79.5      79.5      78.0      80.5
  8         83.0      84.0      83.0      83.0      82.0      84.0
  9         86.0      86.5      86.0      86.0      86.0      87.0
 10         88.5      89.0      89.0      89.0      88.5      90.0
 11         91.0      91.0      91.5      91.5      91.0      93.0
 12         93.0      93.0      94.0      94.0      93.0      95.0
 13         95.0      95.0      96.0      96.0      95.5      97.0
 14         97.0      97.0      98.0      98.0      97.5      99.0
 15         98.5      98.5      99.5     100.0      99.0     101.0
 16        100.5     100.0     101.0     101.5     101.0     103.0
 17        102.0     102.0     102.5     103.0     102.5     104.0
 18        103.5     103.5     104.0     105.0     104.0     106.0
 19        105.0     105.0     105.0     106.5     105.5     107.0
 20        106.5     106.0     106.5     108.0     107.0     108.5
 21        107.5     107.5     108.0     109.0     108.0     110.0
 22        109.0     109.0     109.0     110.5     109.5     111.0
 23        110.0     110.0     110.0     112.0     111.0     112.0
 24        111.0     111.0     111.0     113.0     112.0     113.0
 25        112.0     112.5     112.0     114.5     113.0     114.0
 26        113.0     113.5     113.0     115.5     114.0     115.0
 27        114.0     114.5     114.0     116.5     115.0     116.5
 28        115.0     115.5     115.0     118.0     116.0     117.0
 29        116.0     116.5     116.0     119.0     117.0     118.0
 30        117.0     117.5     117.0     130.0     118.0     119.0
```

```
                          SAMPLE NO 6



RUN NO        1       2       3       4       5       6       7
CURRENT   0.253   0.252   0.252   0.252   0.251   0.251   0.250

TIME                  THERMOCOUPLE OUTPUT

   1       21.0    20.0    21.0    18.0    16.0    19.5    20.5
   2       35.0    35.0    33.0    32.0    31.0    31.5    31.0
   3       44.0    43.0    43.0    41.0    41.0    43.0    41.0
   4       51.0    51.5    50.0    49.0    49.5    50.0    49.0
   5       57.0    57.0    56.0    56.0    50.0    56.0    54.5
   6       62.0    63.0    61.0    61.0    59.0    61.0    59.0
   7       67.0    67.5    65.5    66.0    63.5    65.0    63.0
   8       70.5    70.5    69.0    69.5    67.0    68.5    67.0
   9       73.5    73.5    72.0    72.5    70.5    71.0    69.5
  10       76.5    76.0    74.5    75.0    73.0    74.0    72.0
  11       79.0    78.5    76.5    77.5    76.0    76.0    74.0
  12       81.0    81.0    78.5    79.5    79.0    78.5    76.5
  13       83.5    83.0    80.5    81.5    81.0    80.5    78.0
  14       85.0    85.0    82.5    83.5    83.0    82.0    80.0
  15       87.0    87.0    84.0    85.0    85.0    84.0    82.0
  16       89.0    89.0    86.0    87.0    86.5    85.5    83.5
  17       90.5    91.0    88.0    88.5    88.0    87.0    85.0
  18       92.0    92.5    89.5    90.0    89.5    88.0    86.5
  19       94.0    94.0    91.0    91.5    91.0    89.5    88.0
  20       95.0    96.0    92.5    92.5    92.0    91.0    89.5
  21       96.5    97.0    94.0    94.0    93.5    92.5    91.0
  22       98.0    99.0    95.5    95.0    95.0    93.5    92.0
  23       99.0   100.0    96.5    96.5    96.0    95.0    93.0
  24      100.0   101.5    98.0    97.5    97.0    96.0    94.5
  25      101.0   103.0    99.0    99.0    98.0    97.0    95.5
  26      102.0   104.0   100.0   100.0    99.0    98.0    96.5
  27      103.0   105.0   101.5   101.0   100.0    99.0    97.5
  28      104.0   106.0   102.5   102.0   101.0   100.0    99.0
  29      105.0   107.0   103.5   103.0   102.0   101.0   100.0
  30      106.0   108.0   104.5   103.5   103.0   101.5   101.0
```

```
                         SAMPLE NO 6 (CONTINUED)



RUN NO        8        9       10       11       12       13
CURRENT     0.251    0.251    0.251    0.250    0.249    0.249

TIME                  THERMOCOUPLE OUTPUT

  1          19.0     22.0     24.0     20.0     20.0     16.0
  2          33.5     35.0     35.5     33.5     32.5     32.0
  3          44.0     43.0     44.0     44.0     42.0     41.5
  4          50.0     50.0     51.0     51.0     49.0     49.0
  5          56.0     56.0     55.0     57.0     55.0     55.0
  6          60.0     60.5     59.0     62.0     60.0     60.0
  7          64.0     64.5     62.5     67.0     64.0     64.0
  8          67.0     68.0     66.0     70.5     67.5     67.5
  9          70.0     71.5     69.0     74.0     70.5     71.0
 10          73.0     74.0     72.0     76.0     73.0     73.5
 11          76.0     76.5     75.0     79.0     76.0     76.0
 12          78.5     78.5     77.0     81.0     78.0     78.5
 13          81.0     81.0     79.5     83.0     80.0     80.5
 14          82.5     82.5     81.0     85.0     82.0     82.0
 15          84.0     84.0     83.0     87.0     84.0     84.0
 16          86.0     85.5     84.5     88.5     85.5     86.0
 17          87.5     87.0     86.0     90.0     87.0     87.5
 18          89.0     88.0     87.5     91.5     88.5     89.0
 19          90.0     89.5     89.0     93.0     90.0     90.0
 20          91.5     90.5     90.0     94.0     91.0     91.5
 21          93.0     92.0     91.0     95.5     92.5     92.5
 22          94.0     93.0     92.0     97.0     94.0     94.0
 23          95.0     94.0     93.0     98.0     95.0     95.0
 24          96.0     95.0     94.0     99.0     96.0     96.0
 25          97.0     96.0     95.0    100.0     97.0     97.0
 26          98.0     97.0     96.0    101.0     98.0     98.0
 27          99.0     98.0     97.0    102.0     99.0     99.0
 28         100.0     99.0     97.5    103.0    100.0    100.0
 29         101.0    100.0     98.5    104.0    101.0    101.0
 30         102.0    101.0     99.0    105.0    102.0    102.0
```

Appendix D

Sample Temperature

All values are given in millivolts for copper-constantan thermocouples with a reference junction at 32 F. Numbers 1a and 4a refer to thermocouples imbedded in the surface of sample 1 and sample 4 respectively. All other thermocouples are located near the center of the respective samples.

	Sample Numbers							
	1	1a	2	3	4	4a	5	6
Run No.								
1	0.819	0.822	0.819	0.819	0.809	0.813	0.809	0.809
2	0.856	0.859	0.856	0.856	0.872	0.876	0.872	0.872
3	0.852	0.852	0.852	0.852	0.857	0.863	0.859	0.861
4	0.832	0.837	0.837	0.837	0.859	0.861	0.859	0.859
5	0.850	0.855	0.855	0.852	0.881	0.888	0.881	0.881
6	0.801	0.806	0.805	0.810	0.869	0.871	0.869	0.864
7	0.820	0.830	0.820	0.820	0.871	0.878	0.878	0.878
8	0.850	0.851	0.850	0.850	0.867	0.867	0.867	0.867
9	0.830	0.845	0.836	0.840	0.865	0.870	0.870	0.870
10	0.810	0.804	0.810	0.810	0.853	0.852	0.860	0.865
11	0.820	0.820	0.820	0.814	0.775	0.774	0.775	0.775
12	0.845	0.845	0.845	0.845	0.815	0.816	0.815	0.815
13	0.804	0.812	0.805	0.812	0.792	0.796	0.796	0.796