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Thermal Conductivity Measurements of Porous Materials in Several Gaseous Environments

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Experimental measurements were made of the effective thermal conductivity of two elastomeric heat-shield materials exposed to pure and multicomponent mixtures of gases over a pressure range from 760 to 10^{-6} torr using the line-source technique. Gases and gas mixtures employed were N_2, CO_2, 50% CO_2 and 50% N_2, 50% Ar and 50% CO_2, and 11.6% Ar and 88.4% CO_2, the last combination representing a simulated Martian atmosphere. Data obtained were analyzed extensively to determine causes for the unusual behavior of some of the data sets. The most plausible explanation of the anomalous behavior appeared to be sorption and/or desorption of water vapor. In addition error analyses of the line-source technique of obtaining thermal conductivity and thermal-diffusivity measurements were made which indicated the importance of obtaining accurate measurements of both temperature and heater-wire current, as the errors are of comparable magnitude. Thermal diffusivity measurements using the line-source technique are shown to have inherently greater scatter than thermal conductivity measurements.

Nomenclature

b = y intercept
c_p, c_v = specific heat at constant pressure and volume, respectively
C = Euler's constant = 0.5772157
C_1 = const
e_n = error or uncertainty in an independent variable
e_E = total error in a function involving n independent variables
F = a function of n independent variables
I = current
k = thermal conductivity
m = slope
n = number of variables
q = heat input per length of line-heat source
R = heater wire resistance
t = time
T = temperature (also θ)
x, y, z = spatial coordinates
α = thermal diffusivity
β = $r/2(\alpha t)^{1/2}$
θ = temperature
ρ = density

Introduction

General Description of Present Study

ABLATING materials under consideration for space missions to Mars (Viking program) may be classified as porous solids. The effective thermal conductivity of a porous solid, in addition to depending upon the conductive and radiative properties of the solid, depends upon the conductivity of the gas permeating the solid. Predicting the thermal behavior of an ablative material in a Martian atmosphere is, therefore, dependent upon a knowledge of the Martian atmosphere and the heat-transfer mechanisms in the gas-filled porous solids. Also, a knowledge of the thermal conductivity of such material under vacuum conditions is important, since in many cases the thermal balance of a spacecraft in transit may depend upon a knowledge of the thermal conductivity in vacuum.

Because of these preceding considerations, the first phase of the present study consisted of experimental investigations which were designed to obtain measurements of the effective thermal conductivity of two heat-shield materials at ambient temperature while exposed to pure and multicomponent mixtures of gases over the pressure range of 760 to 10^{-6} torr. Measurements were made at steady-state conditions at each chosen pressure.

The second phase of the present study consisted essentially of an error analysis of thermal-conductivity and thermal-diffusivity measurements obtained by means of the line-source technique. Use of the line-source technique in obtaining thermal-conductivity measurements is relatively recent compared with the guarded hot-plate method, the standard method employed by the National Bureau of Standards. Consequently, the accuracy associated with the technique has not been clearly defined. Thus, an error analysis was believed warranted, especially in light of some unusual and unexpected test results obtained in the first phase of the present study.

Line-Source Technique

The line-source technique of measuring thermal conductivity was first suggested by Schleiermacher[1] in 1888. This method was first applied with success by Van der Held and van Drunen[2] in measuring the conductivity of liquids. The method may also be applied to gases although its greatest usage lies in measurements in solids. Numerous investigators (Refs. 3–10) have utilized the method in relatively recent years, perhaps because of the possibility of obtaining measurements with relative simplicity and speed compared with other steady-state techniques, e.g., the guarded hot-plate method.

The line-source method consists of placing a heater wire (line source of heat) and a thermocouple wire within a sample as indicated in Fig. 1. Upon introducing current into the heater wire, the temperature-time history of the heater wire is recorded and later used to calculate k from the simple expression (derived and discussed later)

$$k = \frac{q}{4\pi(\theta_2 - \theta_1)} \ln \frac{t_2}{t_1} \tag{1}$$

where k = thermal conductivity, q = heat input, θ = temperature, and t = time after initiation of heat generation.

After some simplification and approximation, Eq. (1) is obtained from the exact analytical solution for the temperature-

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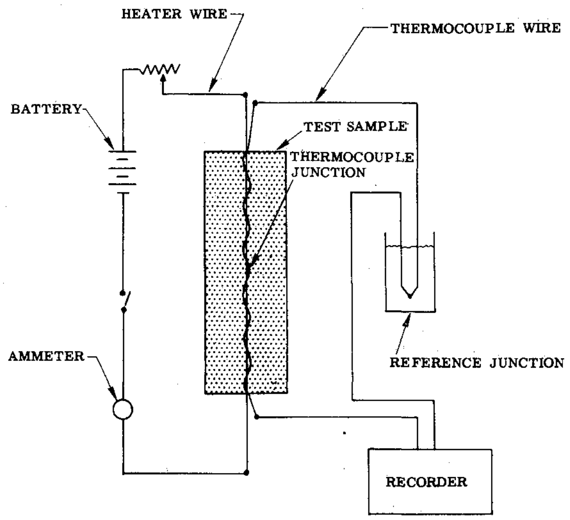

Fig. 1 Schematic diagram of apparatus used for measuring thermal conductivity.

time history in an infinite solid, in which a line-source of heat of infinite length is located. In practice, there are several departures from the ideal conditions analyzed, which may produce unacceptably large inaccuracies in the measured k values. Since an error analysis of the line-source technique does not appear in the literature, one was performed here to evaluate the relative magnitudes of various sources of error.

The line-source technique has also been applied to measurements of thermal diffusivity (α) of solids (Refs. 11 and 12). In this application, an additional temperature-time history is required at a point somewhat removed from the heater wire. Inaccuracies experienced in obtaining thermal-diffusivity measurements have been significantly larger than those of thermal-conductivity measurements ($\pm 20\%$ compared with $\pm 5\%$ for the most accurate measurements). Consequently, there is a need for an error analysis associated with thermal-diffusivity measurements by this technique also.

Heat Transfer in Porous Solids

One of the original purposes of the present study was to obtain thermal-conductivity measurements not only of porous heat-shield materials in a Martian atmosphere, but in various multicomponent mixtures of gases. Such measurements could provide a basis for evaluating methods of predicting conductivity values for any porous solid in any gaseous environment under varying temperature and pressure conditions.

In general, heat transfer through porous solids occurs by the combined mechanisms of gaseous conduction, solid conduction, and radiation. Each of these mechanisms is difficult to model and mathematically describe in a general manner (discussed further in Ref. 13). Consequently, accurate quantitative predictions of the combined effects of conduction and radiation in porous solids under various conditions of temperature and pressure are extremely difficult to obtain from a purely theoretical basis. Some of the most comprehensive efforts along these lines are those of Cheng and Vachon (Refs. 14 and 15).

Experimental Program

General

Thermal conductivity measurements were made of two heat-shield materials using the line-source technique. Measurements were obtained while the materials were exposed to various pure gases and gas mixtures over a range of pressure from atmospheric to 10^{-6} torr.

Materials Investigated

The first material studied is designated SLA-561 and is manufactured by the Martin-Marietta Corp. This material has a nominal density of 12.5 lb/ft^3 and a porosity of approximately 30%. It is a rather complex material composed of a silicone elastomer which is filled with silica spheres, phenolic microballoons, silica fibers, and cork. A complete description of the material, including its ablative properties, can be found in Ref. 16.

The second heat-shield material studied is made by the General Electric Corp. and is denoted as ESM-1004X. This material is chemically foamed and consists of a phenyl-methyl silicone polymer with 12% inorganic aluminum silicate fibers. The material has a nominal density of 15 lb/ft^3 and a porosity of about 90%.

The materials were cast in rectangular blocks of 4 in. by 4 in. by 6 in. A 0.010-in. manganin heater wire and a 30-gage copper-constantan thermocouple wire wrapped about the heater wire, as shown in Fig. 1, were cast into the material during sample preparation. The materials were preconditioned for several weeks at 45% relative humidity in a class-100 clean room prior to testing.

Environmental Condition

The heat-shield samples were exposed to the various environmental conditions in a 24-in.-long, 16-in.-diam high vacuum chamber. The stainless-steel chamber was capable of attaining an operating pressure of 10^{-7} torr. The samples were placed into the chamber through a hinge-mounted door on one end of the chamber. The chamber was equipped with a 360 liter/sec triode ion pump.

Tests were conducted over the pressure range of from atmospheric pressure to 10^{-6} torr using the following pure gases and gas mixture at room temperature: nitrogen, carbon dioxide, argon, 50% CO_2 and 50% N_2, 50% Ar and 50% CO_2, and 11.6% Ar and 88.4% CO_2 (simulated Martian atmosphere). Thermal conductivity values of these gases at atmospheric pressure and 492°R are: CO_2—0.841 (10^{-2}), Ar—0.944 (10^{-2}), N_2—1.40 (10^{-2}) Btu/hr-ft-°F.

Pressures were measured with a pressure gage graduated from 0 to 760 torr in one-torr divisions, a thermocouple gage with a range of 10^{-4} to 20 torr, and an ionization gage with a range of from 10^{-6} to 10^{-4} torr. Since the thermocouple and ionization gages were calibrated in air, correction factors were applied to the indicated readings by using curves supplied by the manufacturer that corrected the indicated pressure to the actual pressure in other gas environments. In the case of gas mixtures, the correction factor applied was an arithmetically weighted average of the correction factor for the constituent gases.

Thermal-Conductivity Measurement System

Heat was generated within the test samples by passing 200 ma of current through the heater wire. The heater circuit consisted of a 6-v battery, an ammeter, and a variable resistor for presetting the heater current prior to initiation of heating the sample. Upon initiating the heater wire current, the temperature-time history of the heater wire was recorded through the use of the thermocouple located on the heater wire, a reference junction, and a pen recorder. A second thermocouple was located $\frac{1}{4}$ in. below the surface and monitored during the tests to determine whether the temperature wave generated within the sample had reached the surface, which would have invalidated the physical conditions upon which the line-source technique is based.

Procedure

After placing a test sample in the environmental chamber, the chamber was pumped to 10^{-6} torr. The test gas or gas mixture was then introduced until atmospheric pressure was reestablished, so that essentially pure gases or gas mixtures filled the chamber. Thermal-conductivity tests were then made at atmospheric and lower pressures. Heater current (200 mm) was applied for a 5 min period, during which the heater-wire temperature and the surface temperature of the sample were recorded.

The chamber was then pumped down to the next lower test pressure and an equilibrium pressure condition was reestablished. After a minimum of 20 min at the new test pressure, during which the sample had also reached thermal equilibrium, the same type of measurements were repeated. When this procedure had been repeated down to the lowest test pressure of 10^{-6} torr, the chamber was refilled with another gas or gas mixture and the entire sequence was repeated.

At this point, it may be appropriate to note that thermal-conductivity measurements of another elastomeric heat-shield material had been conducted in an earlier study by Greenwood and Comparin[17] using the line-source technique. Their results agreed with the NBS values, obtained by the guarded hot-plate method to $\pm 5\%$. Consequently, results presented herein should be interpreted with this range of accuracy in mind since the present study followed essentially the same experimental procedures as those in the cited work.

Values of thermal conductivity of the heat-shield materials were obtained as follows. From the temperature-time traces obtained in each test, a set of temperature-time data points were computer-processed and plotted on a temperature vs ln time basis. The slope of the best-fit (least squares) straight line fitting the points, in accordance with Eq. (1), yielded the thermal-conductivity value.

Experimental Results

Unmodified experimental results

A typical set of experimentally measured values of thermal conductivity over a range of subatmospheric pressures is presented in Fig. 2. These results are typical of porous materials over a range of low pressures, i.e., they are characteristically s-shaped, reflecting a gas phase behavior in accordance with the kinetic theory of gases. These results also illustrate behavior that seems to warrant closer scrutiny of a) experimental test procedures, including the line-source technique itself, and b) data analysis. Specifically, the effective conductivity measured for a CO_2 and Ar mixture (indicated by solid symbols) is greater than that in either a pure CO_2 or pure Ar atmosphere.

The results of Fig. 3 may be explained rather simply if the experimental uncertainty associated with the measurement technique is sufficiently large that the location of the solid symbols is merely a result of natural data scatter. On the other hand, such an explanation would be a rather unlikely one since previous results obtained by the same measurement technique[17] indicated that an accuracy of $\pm 5\%$ should be expected. Consequently, a closer examination of test procedures and data analyses was undertaken and is described in the following section.

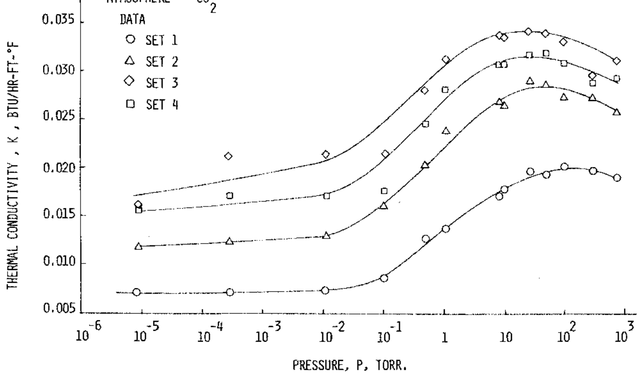

Fig. 2 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

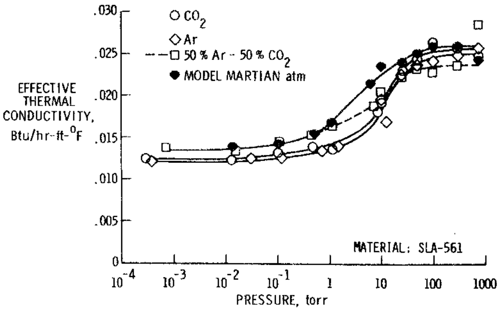

Fig. 3 Thermal conductivity variation of SLA-561 material over a range of pressure in four gaseous environments.

Modified experimental results

a) Recalculation of k values: In employing the line-source technique, it has been observed by numerous investigators that the plot of temperature vs $\ln t$, from which k values are determined, is not linear. This led to the introduction of a t_0 "correction factor," which has the net effect of shifting the origin of the test and thus resulting in a linear variation of temperature as a function of $\ln t$ (Refs. 3–5). Since the computer plots of θ vs $\ln t$ in the present study were not all linear, t_0 correction factors were calculated as in Ref. 18. However, they were found to be of such a small magnitude that measurements beyond the initial stages of the test were essentially unaffected by the "correction" and the nonlinearity remained.

Various possible reasons may be postulated for the nonlinear behavior and require lengthy discussion. Furthermore, in the absence of a further separate experimental program, these possible reasons remain postulatory only. It was thus decided to delete arbitrarily several of the initial data points since the nonlinearity seemed to be concentrated there. Raw data was then reanalyzed and the first 30 sec of data points were deleted. The data was also broken up into three other time segments and reanalyzed. The regions are as follows: 1) includes data points only between 0 and ~30 sec; 2) includes data points only between ~30 and ~120 sec; 3) includes data points between ~120 and 600 sec; and 4) includes data points between ~30 and 600 sec.

Representative results of the modified data for pure CO_2, Ar and their mixture are shown in Figs. 4–8.

b) Test procedure review: In conducting these tests, the vacuum chamber was pumped down to a pressure of 10^{-6} torr and then filled with the test atmosphere to a pressure of 760 torr. After making the first k measurement, the pressure was reduced, thermal equilibrium was allowed to become established, and the procedure was repeated until the lowest test pressure of about 10^{-6} torr was attained. At higher pressures the pump-down process required only a short time period on the order of a few minutes, while at lower pressures a period on the order of several hours was required. There was a minimum time period of 20 min between heating of the sample. Justification for an equilibration period of only 20 min was obtained from Ref. 19, where a period of only several minutes was apparently required for the heat-shield material to attain a new equilibrium thermal conductivity at a new environmental condition. Due to variable time intervals between tests, the possibility that the heat-shield materials had not really attained thermal and chemical equilibrium was considered.

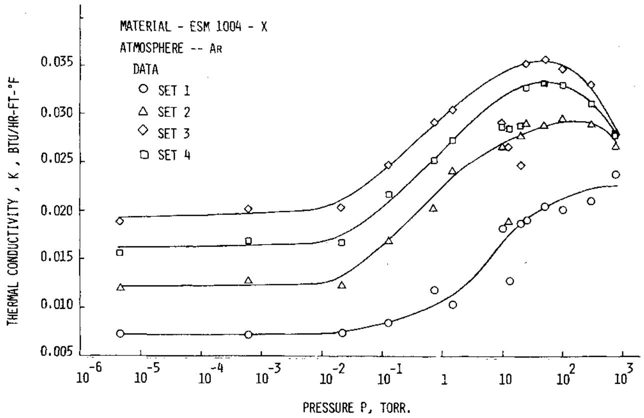

Fig. 4 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

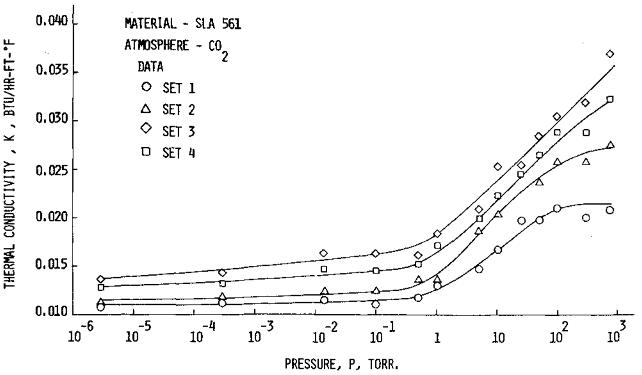

Fig. 6 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

A possible "waiting period" effect was investigated by plotting k vs waiting time for that range of pressures where k was expected to remain constant at pressures higher than the breakaway pressures. No systematic variation of k as a function of waiting period was observed.

The results of Ref. 20 indicate that sorptive processes of gases (Ar, CO_2, N_2, H_2O) in contact with the SLA and ESM materials might not equilibrate for as long as several hundred minutes (as for CO_2 in contact with the SLA material). Using the sorption data presented therein, estimates were made of the amounts of gases sorbed by the samples during the recorded waiting periods. At higher pressures, waiting periods were generally short while the quantities sorbed (moles/gm of sample) were at a maximum, whereas at low pressures the waiting times were long and the quantities absorbed were quite low. In all cases, the amount of gas sorbed by the sample was extremely small compared with sample weight. While sorption processes did no doubt occur, the actual mass of gases affixed or reacting with the sample was very small, leading one to believe that significant changes in k values due to sorption processes should not be expected.

Discussion of Results

Focusing upon results plotted in Figs. 4–8 it is seen that each of the four arbitrary data groupings results in a rather clearly defined curve of thermal conductivity vs pressure. This occurrence is a reflection of the nonlinearity of the temperature-ln-time data obtained. With the exception of a single case, i.e., that of ESM 1004X in a CO_2-Ar mixture at pressures greater than about 100 torr (Fig. 5), all of the conductivity vs pressure curves are at higher levels as the number of data points deleted from the initial stages of the tests is increased. This occurrence is a reflection of the general nature of the nonlinearity of data gathered.

In specific terms, the most meaningful results are those obtained from Sets 3 and 4. Results from Sets 1 and 2 are expository in nature, tending to illustrate a trend, while results from Sets 3 and 4 are more realistic measurements of the actual conductivity of the materials as a function of pressure.

While the trends of the data sets are understandable in light of the observed nonlinearity, it is disconcerting to observe that the nonlinearity of the data is capable of producing rather large variations in the measured conductivity values at a given pressure. Previous investigators have noted nonlinear behavior in the initial stages of test runs and have simply attributed this behavior generally to the finite size of the heater-wire. Several have applied a t_0 correction to eliminate the nonlinearity, which is a superficial but often effective remedy. In the present study, however, the t_0 factor has virtually no effect upon the nonlinear behavior. Consequently, the origins of nonlinear behavior should be explored further.

A multitude of factors that may possibly produce such nonlinearity could be enumerated, among which are finite wire size and edge losses from the heater-wire and sample. The thermocouple materials, method of attachment, conduction

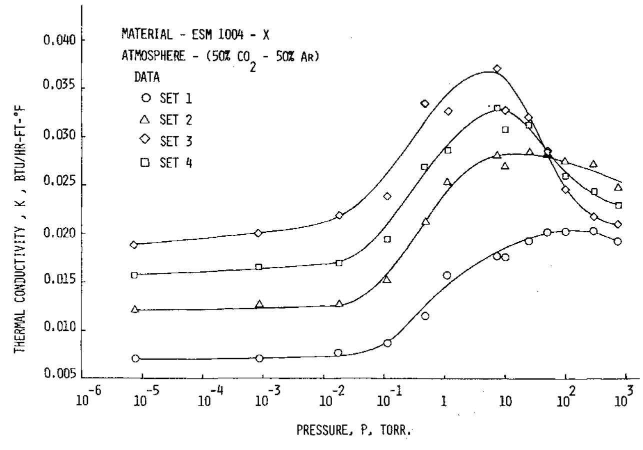

Fig. 5 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

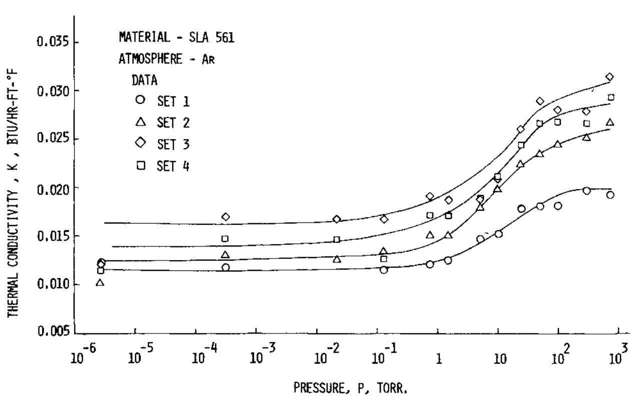

Fig. 7 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

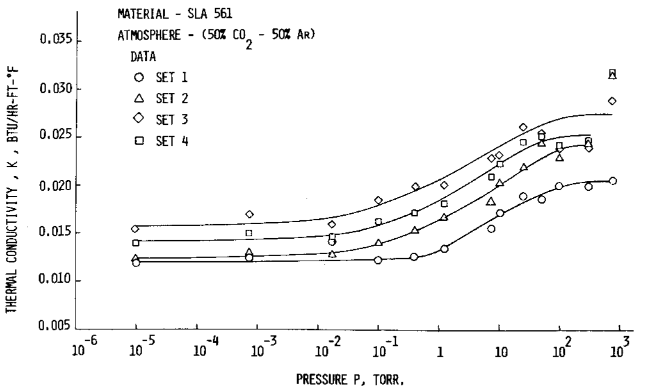

Fig. 8 Variation of thermal conductivity with pressure based upon conductivity values calculated from four different data segments from each test run.

losses, and finite size may also produce errors, in addition to test procedures themselves. Still other sources of error may be hypothesized.

A possible procedures-related factor that may warrant further consideration is the presence of a variable water-vapor content within the sample during the course of the tests. Specifically, the samples were maintained in a 45% relative humidity environment for several weeks prior to testing. However, the samples, during transit and while being installed in the vacuum test chamber, were exposed to an ambient atmosphere that very likely was of higher relative humidity. Griest[20] has reported that the SLA-561 material has a large capacity for water-vapor (about 100 times greater than for CO_2) and furthermore reports that 90% of the sorptive interaction occurs within about 5 min. Wechsler also reports[21] that, in measurements of thermal conductivity of powders at subatmospheric pressures, a considerable scatter of results was found to be caused by a variable water-vapor content. It was also found that a period of days within a vacuum chamber was required to remove water-vapor to a sufficient degree from a sample having a residual water content. In light of the preceding information, the possibility of a variable water-vapor content of a sample affecting the relative conductivity values at different pressures during the testing period should be considered as a possible source of error. At the same time, however, it should be kept in mind that the amount of water-vapor absorbed is extremely small compared with the sample weight, using experimental results from Ref. 20. Without additional experimentation to attempt to isolate such factors, therefore, this explanation must remain speculative in nature.

It is of interest to note several anomalies occurring at the higher test pressures. In many of the tests, it is observed that, in approaching atmospheric pressures (the maximum test pressure), conductivity values have a distinct downward trend. Classically, from the kinetic theory of gases, one would expect a leveling off and constancy of conductivity at pressures higher than the breakaway pressure.

While a drop off in conductivity with increasing pressure is evident in many tests, the reverse trend is also surprisingly present, particularly in the SLA tests, e.g., Fig. 6. The upward trend is even more surprising in that a breakaway pressure is not indicated at all. Perhaps this upward trend is a reflection of a gradual desorption of water-vapor from the solid with the passage of time spent in the vacuum chamber.

A third anomaly is evident in Fig. 5 in that the curves for Sets 3 and 4 not only cross each other at about 70 torr, but also fall beneath the Set 2 curve. This behavior indicates a nonlinear character different from that reflected by the results of all the other tests.

It is conceivable that the foregoing anomalous behavior may be explained by variations of water-vapor content within the sample over the course of test periods. In the absence of quantitative information, however, this can only be speculative.

Additional possible sources of nonlinearity may be lumped into the two categories of 1) experimental errors and inaccuracies, and 2) finite sample effects. Both are investigated and discussed at greater length in the following section.

Error Analysis

Background

In an experiment requiring measurements of several quantities. each of which has an error or "uncertainty" associated with it the total error that propagates into the final result may be estimated by the expression[22]

$$e_E{}^2 = \sum_{n=1}^{i} \left(\frac{\partial F}{\partial n}\right)^2 e_n{}^2 \tag{2}$$

where F is a function of n independent variables having errors e_n. For repeated measurements of the variables, it is assumed that errors are normally distributed about the true value.

The "function" whose resultant error is to be estimated is the thermal conductivity given by Eq. (1). Reviewing briefly the origins of Eq. (1), for an infinite homogeneous solid, in which is immersed an infinitely long line source of heat of zero radius that is instantaneously initiated and maintained at a constant heat generation rate, the transient temperature rise within the solid is given by Ref. 23

$$\theta = \frac{q}{2\pi k} \int_{\beta}^{\infty} \frac{e^{-x^2}}{x} dx \tag{3}$$

where $\beta = r/2(\alpha t)^{1/2}$ and x is a dummy variable.

The integral expression of Eq. (3) may be evaluated by an infinite series, so that the temperature is given by

$$\theta = \frac{q}{2\pi k}\left[\frac{-C}{2} - \ln\beta + \frac{\beta^2}{2(1!)} - \frac{\beta^4}{4(2!)} + \cdots\right] \tag{4}$$

where C = Euler's constant = 0.5772157... For $\beta < 0.16$ the temperature change between two times t_1 and t_2 may be approximated to within 1% by

$$\theta_2 - \theta_1 = (q/4\pi k)\ln(t_2/t_1) \tag{5}$$

The condition that $\beta < 0.16$ is often satisfied by placing the thermocouple junction in contact with the heater wire.

It is seen that Eq. (5) is actually an approximate expression, though it appears that reasonable placement of the temperature measuring device should result in an initial error of less than a maximum of 1%. Other possible sources of error or uncertainty immediately present themselves, however. For example, the specimen or sample size obviously is not infinite. The medium may not be isotropic due to preferential grain orientation arising perhaps because of a casting process. Unless a thermocouple bead is soldered or welded to the heater wire, the value of β is unknown and, furthermore, a hollow portion of the sample in the vicinity of the heater wire is implied. In turn, an uncertainty in the surface resistance between the wire and sample is implied and results physically in perhaps less than ideal diffusion of heat to the solid sample. Depending upon how the thermocouple is attached or unattached, conduction losses along the thermocouple legs may differ. Due to finite sampling size, end effects may be present in the heater wire where the transition is made from a solid to gaseous medium. Finally, the presence of both a heater wire and thermocouple wires violate the model's stipulation of a line heat source of zero thickness. Of course, instrumentation errors inevitably exist in measuring either temperature at a particular time or temperature changes over a time interval. All of the preceding uncertainties may, for purposes of later discussion, be lumped into a single temperature measurement error or uncertainty.

The uncertainty in thermal conductivity may be estimated by applying Eq. (3) to Eq. (1), where $q = I^2R$. One obtains

$$e_k{}^2 = \frac{1}{16\pi^2}\frac{[\ln(t/t_1)]^2}{(\theta-\theta_1)^2}[I^4 e_R{}^2 + 4I^2R^2e_I{}^2] + \frac{q^2}{16\pi^2}\frac{[\ln(t/t_1)]^2}{(\theta-\theta_1)^4}[e_\theta{}^2 + e_{\theta_1}{}^2] + \frac{q^2}{16\pi^2(\theta-\theta_1)^2}[(e_{t_1}{}^2/t_1{}^2) + (e_t{}^2/t^2)] \tag{6}$$

Quantities measured at the initial time value are given the subscript 1, while those measured at any later time are given no subscript.

It will now be informative to substitute numerical values typical of test conditions into Eq. (6). The following values are taken from a typical test run from which thermal conductivity was determined to be 0.032 Btu/hr-ft-°F: $R = 2.85$ ohms/ft (±1%); $I = 0.2$ amps (±2%); $P = I^2R = 0.389$ Btu/hr; $t_1 =$ 5 sec = 0.001389 hr (±1%); $t = 600$ sec = 0.1667 hr (±0.1%); $\theta_1 = 85.37$°F (0.0%, temp. diff. measured); $\theta = 90.00$°F (±0.1%); $k = 0.032$ Btu/hr-ft-°F.

The preceding errors or uncertainties assigned take into account instrumentation accuracy, errors due to lack of resolution, human errors in reading instruments and recorded temperature histories, and general system losses, heat leaks, etc. Using the preceding error estimates the total uncertainty in k, e_k, is found to be 0.513 (10^{-3}).

Comparing this total estimated error to the measured value of k for this run, % error = 16.0%. This is a moderately large percentage of error compared with previously quoted error levels of only ±5% in earlier investigations. This discrepancy may mean that uncertainty limits chosen in this error estimate may have been too large.

The importance of accurate temperature measurements and precision power control are underscored by the numerical values substituted into Eq. (6). That is, in observing the contribution of each of the individual terms of Eq. (6), the following breakdown is obtained: % of total error in k—temperature, 31.6%; power, 68.4%; time, 0.0%.

Summarizing, the foregoing uncertainties produce an uncertainty in the final calculated value of k of ±16%, of which roughly $\frac{1}{3}$ is due to temperature measurement error. If the current control capability is improved to ±1%, or ±0.002 amps, the temperature measurement error is about 50% that of the total error in k. It should also be noted that the preceding error analysis is based upon a determination of k involving only two sets of temperature-time data. The actual determination of k was based upon obtaining the slope of the best-fit (least squares) straight line drawn through many sets of data plotted on an $\ln t/t_0$ vs θ basis. The uncertainty or error associated with the slope determined from several such data points will yield more accurate results than the dual-sample determination.

Thermal Diffusivity

Thermal diffusivity may be determined simultaneously with thermal conductivity using the line-source technique. The procedure, employed by Nix et al., is described in Refs. 11 and 12. In brief, thermal diffusivity is calculated by an iteration process and is based upon the measured values of thermal conductivity and temperature history of the sample.

In order to determine thermal diffusivity, a second thermocouple must be placed within the sample located at a distance sufficiently far from the heater wire to insure that $\beta > 0.16$[11] for purposes of accuracy and convergence. Having measured the temperature-time history of the sample with the diffusivity thermocouple and having calculated a k value based on the heater wire thermocouple measurements for a particular time, a value of β is assumed. The assumed value of β is used to calculate the diffusivity thermocouple temperature using Eq. (4). If the calculated value does not agree with the measured value, a new value of β is assumed. The process is repeated until both temperatures agree. Having achieved convergence, the thermal diffusivity is calculated from the defining equation for β

$$\beta = r/2(\alpha t)^{1/2} = r/2(kt/\rho c_p)^{1/2} \tag{7}$$

Estimating the error associated with measurements of α by this iteration scheme actually comes down to a matter of determining how well the calculated value of temperature from Eq. (4) may be expected to agree with the temperature measured by the diffusivity thermocouple. It is seen from Eq. (7) that β is dependent upon k, r, t, ρ, and c_p; thus, any errors in those quantities will be reflected in the magnitude of β and, consequently, in the calculation of temperature from Eq. (7). In other words, agreement of the calculated and measured temperature values will be only as good as the measurements necessary to evaluate β ultimately. In addition, inaccuracies in the measured value of temperature by the diffusivity thermocouple should also be taken into account.

To begin estimating the error associated with thermal-diffusivity measurements obtained through the use of the line-source technique, the same procedures employed in the preceding section are applied. The calculated temperature is given by Eq. (4) or

$$\theta = (q/2\pi k)f(\beta) \tag{8}$$

from which

$$f(\beta) = (2\pi k/q)\theta \tag{9}$$

The uncertainty in $f(\beta)$ is given by

$$\frac{\omega_{f(\beta)}}{f(\beta)} = \left[\left(\frac{\partial f(\beta)}{\partial k}\frac{\omega_k}{f(\beta)}\right)^2 + \left(\frac{\partial f(\beta)}{\partial \theta}\frac{\omega_\theta}{f(\beta)}\right)^2 + \left(\frac{\partial f(\beta)}{\partial q}\frac{\omega_q}{f(\beta)}\right)^2\right]^{1/2} \tag{10}$$

Evaluating the partial derivatives one obtains

$$\frac{\omega_{f(\beta)}}{f(\beta)} = \left[\left(\frac{\omega_k}{k}\right)^2 + \left(\frac{\omega_\theta}{\theta}\right)^2 + \left(\frac{\omega_q}{q}\right)^2\right]^{1/2} \tag{11}$$

Now since α is obtained from β, it is necessary to ask how the uncertainty associated with $f(\beta)$ is related to that associated with β itself. Recall that

$$\begin{aligned} f(\beta) &= -\frac{C}{2} - \ln\beta + \frac{\beta^2}{2.1!} - \frac{\beta^4}{4.2!} + \cdots \\ &= -\frac{C}{2} - \ln\beta + \sum_{n=1}^{\infty}\frac{\beta^{2n}}{2n\cdot n!}(-1)^{n+1} \end{aligned} \tag{12}$$

The uncertainty in $f(\beta)$ is

$$\omega_{f(\beta)} = [\partial f(\beta)/\partial\beta]\omega_\beta \tag{13}$$

From Eqs. (12) and (13)

$$\omega_{f(\beta)} = \left[-\frac{1}{\beta} + \sum_{n=1}^{\infty}\frac{\beta^{2n-1}}{n!}(-1)^{n+1}\right]\omega_\beta \tag{14}$$

Solving for ω_β

$$\omega_\beta = \frac{\omega_{f(\beta)}}{\left[\frac{-1}{\beta} + \sum_{n=1}^{\infty}\frac{\beta^{2n-1}}{n!}(-1)^{n+1}\right]} \tag{15}$$

It is seen that $\omega_\beta > \omega_{f(\beta)}$ for

$$\left[-\frac{1}{\beta} + \sum_{n=1}^{\infty}\frac{\beta^{2n-1}}{n!}(-1)^{n+1}\right] < 1 \tag{16}$$

or

$$\sum_{n=0}^{\infty}\frac{\beta^{2n-1}}{n!}(-1)^{n+1} < 1 \tag{17}$$

For small values of β (large time values) or for β values corresponding to a 1-sec time increment, the preceding series will be less than one. Thus, for most practical time values, the uncertainty in β will always be greater than the uncertainty in $f(\beta)$. This may explain the normally higher uncertainty (data scatter) associated with thermal-diffusivity measurements using the line-source technique.

Summary

In the experimental phase of the present study the temperature vs ln time data obtained, from which thermal conductivity values were calculated, was of a nonlinear character. A t_0 correction factor applied to the data had no significant effect on the nonlinearity. Variable waiting periods between tests appeared to have little systematic effect upon the thermal conductivity results obtained.

Several instances of anomalous behavior were present in the conductivity results obtained at higher pressures for many of the combinations of solid material and gas mixture. While it seemed improbable that such anomalies were attributable to sorption of Ar, CO_2, or N_2, it was suggested that sorption (or desorption) of water vapor could conceivably result in such apparently anomalous behavior. In the absence of quantitative measurements, however, such possible explanations must remain speculative in nature.

An error analysis of the line-source technique of measuring thermal conductivity was performed. Results indicate that the major sources of error in determining the thermal conductivity are associated with temperature measurement errors and uncertainties in the constancy of power supplied by the heater wire. Expressions are presented for estimating the accuracy of conductivity values obtained from multiple sets of temperature-ln time data.

An error analysis of obtaining thermal diffusivity measurements utilizing the line-source technique was also performed. Results indicate that the percent error is inherently larger than that associated with thermal conductivity measurements, also obtained using the line-source technique.

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