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**THERMAL CONDUCTIVITY OF
CARBON FOAM**

J. D. Lore
H. L. Richards
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September 1974

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CARBIDE**

OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

*prepared for the U.S. ATOMIC ENERGY COMMISSION
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ABSTRACT

Carbon foam materials, carbonized to 1273 K and graphitized to 3273 K prior to testing, were evaluated for thermal conductivity by three steady-state techniques: guarded hot plate, comparative apparatus, and radial flow. Tests were conducted over the temperature interval from 300 to 2800 K. Analyses were made of the material fabrication parameters, pertinent physical properties, and employed test techniques. Interpretation of data and a comparison with the findings of other laboratories were accomplished.

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SUMMARY

Carbon foam, a cellular material of low density and relatively high compressive strength, is employed for load bearing and insulative aerospace applications. The thermophysical character of carbon foam, relatively obscure (particularly at high temperatures), is of interest for design and engineering applications. Carbon foam materials, carbonized to 1273 K and graphitized to 3273 K prior to testing, were evaluated for thermal conductivity in a nitrogen environment from 300 to 2800 K by several overlapping techniques. Continuous functions of temperature for thermal conductivity were determined, and comparisons were made with the findings of other laboratories for similar materials.

INTRODUCTION

Carbon foam is a rigid, cellular material consisting of carbonized polyurethane-polyfurfuryl alcohol mixtures. Processed castings have been employed as a structural and insulation material in high-temperature applications. Carbon foam exhibits an excellent strength-to-weight ratio, a superior thermal stability compared to organic foam systems, a low thermal conductivity, and a low linear coefficient of thermal expansion. The material (relatively new, unique, and potentially useful in aerospace applications) has been prepared by several investigators⁽¹⁻⁴⁾ and described in some detail.^(5,6) The material's thermophysical character, a primary consideration in insulative applications, is virtually undocumented. The temperature dependency of thermal conductivity over the 300 to 2800 K interval is the subject of this report.

DETERMINING THE THERMAL CONDUCTIVITY OF CARBON FOAM

MATERIALS

Fabrication Process

Carbon foam material, typical of recent production formulations, was prepared from a two-component rigid urethane system to which polymerized turturyl alcohol was added.⁽⁸⁾ The exact formulation depends upon the desired product density. Cylindrical test billets, 250 mm in diameter by 430 mm long, were cast by pouring the organic-aqueous mixture into an aluminum mold at 323 K. The foaming process and rising of the casting was completed in the mold to produce the so-called "green" billet which was cured at room temperature for 10 to 15 days. Converting the green castings to carbon foam was carried out in furnaces having an inert (nitrogen) atmosphere. Two castings were produced, as just described, tailoring the manufacturing parameters to produce nominal 220 kg/m³ material. One casting was carbonized to 1273 K after a 60-hour treatment, another was graphitized to 3273 K after a 10-day treatment. Both were sectioned for physical properties testing.

Pertinent Physical Properties

Porosimetry analyses were conducted on both the carbon foam billet heat treated to 1273 K for "low-fired" (LF) material and also on the casting treated to 3273 K or "high-fired" (HF) material. The findings are summarized in Table 1.

Table 1
PERTINENT PHYSICAL PROPERTIES OF CARBON FOAM BASED
ON POROSIMETRIC ANALYSES

Physical Property ⁽¹⁾	1273 K Heat Treatment (low fired)		3273 K Heat Treatment (high-fired)	
	Mean Value ⁽²⁾	Standard Deviation, σ ⁽³⁾	Mean Value ⁽²⁾	Standard Deviation, σ ⁽³⁾
Sample Weight (g)	0.0519	0.0140	0.0534	0.004
Apparent Density ⁽⁴⁾ (kg/m ³)	210	40	260	20
Bulk Density ⁽⁵⁾ (kg/m ³)	387	166	400	35
Real Density ⁽⁶⁾ (kg/m ³)	1590	307	1670	82
Porosity less than 10 μ m (%)	21.42	22.63	18.99	10.24
Total Open Porosity (%)	77.73	10.05	75.91	2.67
Surface Area (m ² /g)	3.197	3.322	2.486	2.203
Volume-Average Equivalent Pore Diameter ⁽⁷⁾ (μ m)	44.23	12.90	39.44	11.21

(1) Measurements obtained using the Aminco porosimeter.

(2) Average for 26 observations on samples extracted from locations uniformly distributed throughout a 250-mm-diameter, 430-mm-long cylindrical billet.

(3) $\sigma = \sqrt{\frac{n \sum v_i^2 - (\sum v_i)^2}{n(n-1)}}$, where "v" represents the measured value and "n" the number of observations, n = 26.

(4) Based on the sample weight prior to testing.

(5) Obtained by infiltration of all surface pores greater than 300 μ m in diameter.

(6) Value for the carbon matrix.

(7) Median value for both cellular axes, based on the pore volume.

Large variations in the surface area and pore diameter throughout each billet were evidenced, and other physical dissimilarities may be noted; but, for test purposes, the LF and HF materials are herein considered as roughly equivalent except for the extent of heat treatment. It is felt that the 3273 K treatment (ie, extensive graphitization) gave rise to the major porosimetric differences between the LF and HF materials.

MEASUREMENT TECHNIQUES

Guarded Hot Plate

A guarded hot plate (GHP)(7) (Dynatech Model TCFGM-N18) was employed for the low-temperature (300 - 1000 K) measurements of thermal conductivity. The instrument test section, as depicted in Figure 1, consists of an inner (main) heater surrounded by an annular guard heater. Function of the guard heater is to eliminate radial heat losses and to force the heat generated in the main heater to flow in one direction through the two test samples. Auxiliary outer guard heaters are inserted between the samples (203-mm-diameter by 25-mm-thick solid cylinders) and the heat sinks. In this fashion, the sample temperature can be raised to any value merely by adjusting the power to the auxiliary heaters. For tests above 700 K, a cylindrical outer guard heater is placed around the entire test section to minimize radial heat losses and to facilitate control of the guard section of the hot plate. The entire test assembly is surrounded by a liquid-cooled shroud, and the space between the shroud and test stack is filled with insulating powder.

Figure 1. TEST SECTION OF THE GUARDED HOT PLATE.

When the test section has reached thermal equilibrium, the temperature difference across the two samples and the electrical input to the main heater are measured and used to calculate the thermal conductivity of the sample material as follows:

$$\lambda_{avg} = \frac{Q_{total}}{A} \left| \frac{1}{\left(\frac{\Delta T}{\Delta \ell}\right)_{top\ sample} + \left(\frac{\Delta T}{\Delta \ell}\right)_{bottom\ sample}} \right| \quad (1)$$

where:

λ_{avg} represents the average thermal conductivity of the two samples (watts/m - K),

Q_{total} the total input to the main heater (watts),

A the cross-sectional area of the main heater (m²),

ΔT the temperature difference across the sample (K), and

$\Delta \ell$ the sample thickness (m).

Conductivities ranging from 0.02 to 2 watt/m - K have been successfully measured.⁽⁸⁾

All measurements on carbon foam were obtained in a nitrogen environment at a pressure slightly positive (1.7 kPa) with respect to the ambient surroundings. Measurements were made over the temperature interval from 300 to ~ 1000 K.

Comparative Apparatus

A comparative-apparatus (CA) method has been employed for insulation materials as a secondary means for measurement which is especially useful for diminutive specimens.^(9,10) The method is essentially a compromise for materials with intermediate thermal conductivities (0.2 to 100 watt/m - K), but it overlaps the GHP range and was applied as a supporting analysis for carbon foam. A drawing of the test section is presented in Figure 2. The sample geometry was a solid cylinder (25 mm in diameter by 64 mm thick) which was sandwiched between two similar samples of a standard reference material (Pyroceram Code 9606). Heaters above and below the samples establish the temperature gradients and overall temperatures. Radial losses from the stack are avoided by controlling the temperature profile down an outer guard tube so that it matches the gradient in the composite sample stack. At equilibrium, assuming that the heat flow along the length of the stack is uniform and constant, the conditions for measurement are met.

Figure 2. TEST SECTION OF THE COMPARATIVE APPARATUS.

The temperature difference (ΔT) is related to the amount of heat, q (watt), according to Fourier's heat conduction equation:⁽¹¹⁾

$$\Delta T = \frac{\Delta \ell}{\lambda} \left(\frac{q}{A} \right) \quad (2)$$

where:

$\Delta \ell$ represents the distance between the two temperature-measuring points,

A the area of the cross section perpendicular to the direction of heat flow, and

λ the coefficient of thermal conductivity.

It is quite obvious from Equation 2 that when the quantity (q/A) is kept the same for two materials—one of known thermal conductivity ($\lambda_{\text{reference}}$) and one of unknown thermal conductivity (λ_{sample})—the respective temperature drops are related as follows:

$$\Delta T \left(\frac{\lambda}{\Delta \ell} \right)_{\text{sample}} = \Delta T \left(\frac{\lambda}{\Delta \ell} \right)_{\text{reference}} \quad (3)$$

Thus, by measuring the temperature difference between the two thermocouples a distance $\Delta \ell$ apart in both the reference material and test sample, the unknown thermal conductivity is:

$$\lambda_{\text{sample}} = \left(\frac{\lambda \Delta T}{\Delta \ell} \right)_{\text{reference}} \times \left(\frac{\Delta \ell}{\Delta T} \right)_{\text{sample}} \quad (4)$$

The condition of equal heat flux [or heat-flow density (q/A)] is obtained by placing a test sample and reference material of equal cross section in intimate contact with each other and by holding the two between a heater and a heat sink (Figure 2). Tests on carbon foam were conducted in a nitrogen environment at a positive pressure of approximately 1.7 kPa over the temperature interval from 400 to ~ 1100 K.

Radial Outflow

High-temperature measurements of thermal conductivity were acquired using a radial-flow (RF) apparatus constructed after the design of Wagner⁽¹²⁾ as LASL. The technique and its application to carbonaceous material have been fully described.⁽¹³⁾ The primary reported use of the LASL instrument has been to test graphites for nuclear rocket and reactor applications.^(14 - 16)

This report details the initial application of the radial outflow technique to a material such as carbon foam. A functional description of the apparatus has been made in order to clarify this "nonstandard" test application, but much of the procedural detail should be attributed to the original investigator.^(12,13)

An overview of the high-temperature thermal conductivity testing facility and a schematic of the employed furnace are provided in Figures 3 and 4. The RF apparatus is located in the central area just to the left of the control panel (Figure 3) which contains potentiometric controls for the 50 kVA, three-phase, DC power source (Miller SR-1000-A7) which supplies the resistively heated, water-cooled furnace. Current is passed through a solid cylinder (12.7 mm in diameter by 165 mm long) shown in Figure 5 (far right). The adjacent specimen-to-heater rod assembly is accomplished by rod insertion into the hollow cylindrical specimen (12.8 mm ID by 25.4 mm OD by 89.0 mm long), as illustrated. The assembly is then secured in the furnace, as viewed in Figure 6, having been press fitted into the graphite (Grade ATJ) grips which are connected to the positive and negative electrode terminal feedthroughs. The water-cooled electrodes receive power directly from the supply which is connected by flat-faced bus bars and electrical cables (Size 1 wire). Measurement of the applied power requires a voltage drop measurement (v) over the middle 17.0 mm length (ℓ) of the specimen. This measurement is accomplished by employing two pointed graphite voltage probes (Grade UF4S) which make contact at the heater rod surface passing through the specimen.

Figure 3. HIGH-TEMPERATURE TESTING FACILITY.

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The probes are supported in the furnace wall and are fed through to electrical leads and inputted to an integrating-type digital voltmeter (Figure 6). Current (i) is measured by inserting a precision air-cooled shunt in series with the power train and measuring the voltage drop across the shunt. Energy supplied to the specimen by the rod instills a radial-outflow condition. The outer surface of the test specimen loses energy to the surrounding graphite-coated (Dylon AE), cold, black furnace wall predominantly by radiation and, thus, a temperature gradient is set up through the wall of the hollow cylinder.

Figure 4. BOX-FURNACE, RADIAL-HEAT-FLOW, THERMAL-CONDUCTIVITY ARRANGEMENT.

Figure 5. HIGH-TEMPERATURE THERMAL CONDUCTIVITY TEST SPECIMENS.

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Equation 2 may be restated in terms of thermal energy, Q (joules), as:

$$Q = \lambda A t \frac{\Delta T}{\Delta s}, \quad (5)$$

where:

t represents the transport time (s),

A the area measured at right angles to the direction of heat flow for this condition (m^2), and

ΔT the difference in temperature (k) between two parallel surfaces a distance, Δs , apart.

Figure 6. RADIAL-FLOW TEST SECTION.

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A general equation for steady, one-dimensional (radial) flow can be inferred from Equation 5 as:

$$\frac{dQ}{dt} = -\lambda A \frac{dT}{ds} \quad , \quad (6)$$

where the minus sign is introduced because the temperature decreases in the direction of heat flow.

Further:

$$\frac{dQ}{dt} = i_v = P \text{ (watt)} \quad , \quad (7)$$

where P is a constant under steady-state conditions.

For a hollow cylinder, as depicted in Figure 7, the cylindrical shell has a surface area represented by:

$$A = 2\pi r \ell \quad , \quad (8)$$

where ℓ represents the included axial distance (m) and $r = s$ = the transport distance (m). Substituting for s and A in Equation 6 gives:

$$\frac{Pdr}{r} = -2\pi\lambda\ell dT \quad . \quad (9)$$

Integration between limits will yield an expression for the thermal conductivity (λ):

$$\lambda = \frac{P\ell n \frac{r_2}{r_1}}{2\pi\ell(T_1 - T_2)} \quad . \quad (10)$$

Equation 10 was employed to compute the radial flow λ values.

Four 0.76-mm-diameter pyrometer sighting holes were drilled at the approximate centerline of the gage section to the depths indicated in Figure 7. This geometrical configuration facilitated four observations of temperature (T_1, T_2, T_3, T_4) at each furnace power setting at four incremental radial sites (r_1, r_2, r_3, r_4) extending outward from the cylindrical specimen's axis. Temperature measurements were obtained using a single, disappearing, filament-type, micro-optical pyrometer (Pyro 95) operating at the nominal red wavelength (650 nm) and calibrated with a black body source. Responses were corrected for the intervening media (quartz window and the ambient nitrogen test environment) at 1.7 kPa.

RESULTS

Test Objectives, Parameters, and Findings

Employment of materials such as carbon foam for load bearing and insulative aerospace applications is dependent on the combination of a low thermal conductivity, inertness at high temperatures, a low density and chemical compatibility with other carbon-based materials. These requirements necessitate a thermophysical characterization of candidate materials from room temperature to their destruction temperature.

Test specimens of the low and high-fired carbon foam were evaluated for thermal conductivity by the techniques just described. All measurements were made in a direction perpendicular to the axis of cell rise, as indicated in Figure 8—a cell cross section depicted somewhat simplistically as an ellipsoid. Tests were conducted in a nitrogen environment at +1.7 kPa. Thermal-conductivity results for the nominal 220 kg/m³ carbon foam are compiled in Tables 2 and 3 and are illustrated in Figures 9 - 11.

Reduction and Analysis of Data

Thermal conductivity values, as given in Tables 2 and 3, are based on the aggregation and reduction of GHP, CA, and RF results, considered separately. Data compiled therein are represented both as observations and as values computed by regression analyses. Continuous thermal conductivity functions of temperature are also tabulated as an empirical means of generating average values for comparison at a given temperature.

These computed values for the GHP technique were at variance with the observed values by as much as 7.3% for the LF material and as little as 1.8% for the HF material. Thus, the empirical least-squares analyses of GHP results gave the least and most precise mathematical

Figure 7. HOLLOW, CYLINDRICAL, RADIAL-FLOW TEST SPECIMEN.

models of all employed test techniques. Data scatter for the CA results was much more consistent—2.8% and 4.4% for the HF and LF material, respectively. The RF technique gave results deviant, on the average, by 1.9% and 3.8% for the HF and LF foam.

Figure 8. ONE-DIMENSIONAL REPRESENTATION OF A CARBON FOAM CELL.

GHP, CA, and RF data were compiled and reduced by regression analyses giving each observation equal statistical weight to produce a comprehensive thermal conductivity/temperature function. Summary results for the LF and HF carbon foam are

Table 2
COMPARISON OF THERMAL-CONDUCTIVITY RESULTS
FOR LOW-FIRED CARBON FOAM

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ	Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\bar{\lambda} \times 10^2$	Correlation Coefficient, r (4)
		Observed(1)	Calculated(2)				
Guarded	324	0.173	0.167	$\lambda = a_1 T + a_0$ $a_1 \times 10^4$ $a_0 \times 10^2$ 3.98 3.84	0.021	7.3	0.937
Hot Plate	365	0.190	0.183				
	373	-	0.186				
	469	0.228	0.224				
	473	-	0.262				
	530	0.275	0.249				
	573	-	0.266				
	574	0.236	0.266				
	669	0.295	0.304				
	673	-	0.306				
	675	0.298	0.306				
	763	0.315	0.342				
	773	-	0.345				
	779	0.337	0.348				
	871	0.421	0.384				
	979	0.434	0.428				
Comparative	476	0.307	0.285	2.76 15.4	0.016	4.4	0.927
Apparatus	524	0.292	0.298				
	539	0.325	0.302				
	573	0.316	0.312				
	616	0.308	0.323				
	661	0.326	0.336				
	673	-	0.339				

(1) Value based on an individual observation.

(2) Value computed by regression analysis.

(3) $\sigma = \left| \frac{(\sum \lambda_{\text{obs}} - \sum \lambda_{\text{calc}})^2}{i-p-1} \right|^{1/2}$, where "i" represents the number of data pairs and "p" the degree of the polynomial function.

(4) $r = \left| \frac{[a_1 \sum (T_i - \bar{T})(\lambda_i - \bar{\lambda}) + a_2 \sum (T_i - \bar{T})^2 (\lambda_i - \bar{\lambda}) + \dots + a_p \sum (T_i - \bar{T})^p (\lambda_i - \bar{\lambda})]}{\sum (\lambda_i - \bar{\lambda})^2} \right|^{1/2}$

Table 2 (Continued)

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ	Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\bar{\lambda} \times 10^2$	Correlation Coefficient, r (4)
		Observed(1)	Calculated(2)				
Comparative Apparatus	721	0.378	0.352				
	753	0.388	0.361				
	773	-	0.367				
	810	0.403	0.377				
	843	0.379	0.386				
	873	-	0.394				
	898	0.412	0.401				
	927	0.388	0.409				
	957	0.440	0.417				
	973	0.428	0.422				
	1022	0.434	0.435				
	1073	-	0.450				
	1113	0.473	0.460				
	1141	0.471	0.468				
	1173	0.492	0.477				
	1187	0.460	0.487				
Radial Flow	1200	0.788	0.795	$\lambda = a_4 T^4 + a_3 T^3 + a_2 T^2 + a_1 T + a_0$	0.028	1.9	0.997
	1273	-	0.802	$a_4 \times 10^{13} \quad a_3 \times 10^9 \quad a_2 \times 10^6 \quad a_1 \times 10^2 \quad a_0$			
	1391	0.853	0.839	3.75 -2.78 8.11 -1.00 5.19			
	1473	0.863	0.878				
	1523	-	0.908				
	1609	0.973	0.965				
	1661	0.992	1.00				
	1726	1.05	1.05				
	1773	-	1.09				
	1814	1.16	1.13				

Table 2 (Continued)

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ	Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\lambda \times 10^2$	Correlation Coefficient, r (4)
		Observed(1)	Calculated(2)				
Radial Flow	1867	1.19	1.18				
	1923	1.24	1.23				
	1979	1.32	1.29				
	2023	-	1.33				
	2064	1.33	1.38				
	2149	1.46	1.47				
	2191	1.51	1.52				
	2273	-	1.63				
	2337	1.72	1.72				
	2379	1.81	1.78				
	2459	1.93	1.93				
	2523	-	2.03				
	2593	2.15	2.16				
	2685	2.43	2.37				
	2768	2.50	2.59				
	2773	-	2.60				
	2806	2.73	2.69				

Table 3
COMPARISON OF THERMAL-CONDUCTIVITY RESULTS
FOR HIGH-FIRED CARBON FOAM

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ	Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\lambda \times 10^2$	Correlation Coefficient, r (4)
		Observed (1)	Calculated(2)				
Guarded	307	1.19	1.19	$\lambda = a_3 T^3 + a_2 T^2 + a_1 T + a_0$ $a_3 \times 10^9 \quad a_2 \times 10^5 \quad a_1 \times 10^3 \quad a_0$ 4.95 -1.08 7.89 -0.361	0.026	1.8	0.970
Hot Plate	320	1.20	1.22				
	337	1.26	1.27				
	346	1.30	1.29				
	371	1.35	1.34				
	373	-	1.34				
	380	1.39	1.36				
	399	1.39	1.39				
	409	1.39	1.40				
	421	1.37	1.42				
	424	1.46	1.43				
	473	1.50	1.48				
	478	1.52	1.49				
	501	1.52	1.52				
	573	-	1.56				
	577	1.55	1.56				
	600	1.53	1.57				
	672	1.59	1.59				
	673	-	1.58				
	773	1.63	1.60				
	873	-	1.62				
	874	1.62	1.63				
	973	-	1.69				
	986	1.71	1.71				

(1) Value based on an individual observation.

(2) Value computed by regression analysis.

$$(3) \sigma = \left[\frac{(\sum \lambda_{\text{obs}} - \sum \lambda_{\text{calc}})^2}{i - p - 1} \right]^{1/2}, \text{ where "i" represents the number of data pairs and "p" the degree of the polynomial function.}$$

$$(4) r = \left[\frac{[a_1 \sum (T_i - \bar{T})(\lambda_i - \bar{\lambda}) + a_2 \sum (T_i - \bar{T})^2 (\lambda_i - \bar{\lambda}) + \dots + a_p \sum (T_i - \bar{T})^p (\lambda_i - \bar{\lambda})]}{\sum (\lambda_i - \bar{\lambda})^2} \right]^{1/2}$$

Table 3 (continued)

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ				Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\bar{\lambda} \times 10^2$	Correlation Coefficient, r (4)
		Observed(1)	Calculated(2)							
				$a_3 \times 10^9$	$a_2 \times 10^5$	$a_1 \times 10^5$	a_0			
Comparative Apparatus	395	1.61	1.55	1.51	-0.429	4.03	0.531	0.049	2.8	0.677
	404	1.47	1.56							
	436	1.56	1.60							
	457	1.70	1.62							
	473	-	1.63							
	483	1.59	1.65							
	514	1.75	1.69							
	545	1.65	1.70							
	573	-	1.71							
	607	1.68	1.73					0.076	3.8	0.975
	637	1.79	1.75							
	673	-	1.76							
	697	1.80	1.77							
	728	1.74	1.77							
	756	1.80	1.78							
	773	-	1.78							
	789	1.79	1.78							
	815	1.79	1.78							
	846	1.75	1.78							
	873	-	1.78							
	905	1.79	1.78							
	934	1.74	1.78							
	923	-	1.78							
	995	1.81	1.78							
	1025	1.84	1.78							
	1055	1.74	1.78							
	1073	-	1.78							
	1117	1.74	1.78							

Table 3 (continued)

Method	Mean Temperature, T (K)	Thermal Conductivity, λ (watt/m-K)		Polynomial Function of T for λ	Standard Deviation of Fit, σ (3)	Percent Deviation, $\sigma/\bar{\lambda} \times 10^2$	Correlation Coefficient, r (4)
		Observed(1)	Calculated(2)				
Radial Flow	1023	-	1.48	$\lambda = a_4 T^4 + a_3 T^3 + a_2 T^2 + a_1 T + a_0$ $a_4 \times 10^{13}$ $a_3 \times 10^9$ $a_2 \times 10^5$ $a_1 \times 10^2$ a_0 -5.69 5.24 -1.44 1.70 -1.59			
	1082	1.67	1.58				
	1185	1.64	1.66				
	1235	1.68	1.69				
	1273	-	1.65				
	1305	1.71	1.72				
	1359	1.85	1.74				
	1423	1.73	1.77				
	1472	1.83	1.78				
	1523	-	1.71				
	1605	1.79	1.83				
	1667	1.87	1.86				
	1733	1.88	1.90				
	1773	-	1.79				
	1809	2.00	1.94				
	1863	1.97	1.99				
	1911	1.98	2.03				
	1967	2.06	2.09				
	2023	-	1.98				
	2051	2.29	2.18				
	2116	2.30	2.27				
	2178	2.37	2.36				
	2259	2.53	2.48				
	2273	-	2.29				
	2332	2.52	2.60				
	2405	2.55	2.73				
	2443	2.73	2.79				
	2502	3.02	2.90				
	2523	-	2.66				
	2605	3.19	3.07				
	2773	-	3.00				
	2776	3.29	3.33

Figure 9. THERMAL CONDUCTIVITY OF LOW-FIRED CARBON FOAM (STANDARD 90% TL).

Figure 10. THERMAL CONDUCTIVITY OF HIGH-FIRED CARBON FOAM (STANDARD 90% TL).

Figure 11. COMPARISON OF THERMAL CONDUCTIVITY FOR LOW-FIRED, ①, AND HIGH-FIRED, ②, CARBON FOAM.

compiled in Table 4. All observations, the computed trend line, and 90% tolerance limits are displayed in Figures 9 and 10 wherein the separate techniques are differentiated symbolically. As indicated in Table 4, the percent deviation of the composite CA, GHP, and RF data about the computed regression lines are, respectively, 8.1 and 4.7% for the LF and HF materials. A comparison of the LF and HF results is provided in Figure 11. HF results were higher by approximately 320% at 298 K, 165% at 1273 K, 79% at 1773 K, and 46% at 2523 K.

Consideration of the results given in Tables 2 and 3 and depicted in Figures 9 and 10 shows good agreement between the GHP and CA results within the 400 - 1000 K overlap region and reasonable compatibility between the aggregate CA/GHP and RF data groupings, considering that in the 1000 to 1300 K interfacial region each technique manifests the highest levels of measurement error. Specifically, the CA, GHP, and RF techniques (respectively) suffered in this region due to thermocouple noise/heat losses and large absolute pyrometric errors. These effects do bias the data points lower for the CA/GHP and higher for the RF methods; but, when all data sets are aggregated (Table 4), the effect upon the composite regression line is minimized.

Interpretation

Individual GHP and CA thermal conductivity values were obtained on the basis of multiple pairs of temperature determinations; whereas, each RF value was based on two pyrometer readings which defined the temperature gradient. GHP and CA thermal transport distances

Table 4
REPRESENTATIVE THERMAL CONDUCTIVITY RESULTS FOR CARBON FOAM
(Composite of All Methods: GHP, CA, and RF)

Previous Heat Treatment Temperature (K)	Mean Temperature, T (K)	Computed Thermal Conductivity, λ (watt/m-K)	Polynomial Function of T for λ (1)					Standard Deviation of Fit, σ (2)	Percent Deviation, $\sigma/\bar{\lambda} \times 10^2$	Correlation Coefficient, r (3)
1273 (low fired)	298	0.290	$\lambda = a_4 T^4 + a_3 T^3 + a_2 T^2 + a_1 T + a_0$					0.055	8.1	0.991
	523	0.274	$a_4 \times 10^{13}$	$a_3 \times 10^{10}$	$a_2 \times 10^6$	$a_1 \times 10^3$	a_0			
	773	0.374	1.17	-6.69	1.62	-1.08	0.485			
	1023	0.481								
	1273	0.653								
	1523	0.851								
	1773	1.07								
	2023	1.33								
	2273	1.64								
	2523	2.04								
3273 (high fired)	2773	2.58						0.084	4.7	0.956
	298	1.22	-3.73	-18.0	-2.25	4.28	0.198			
	523	1.61								
	773	1.75								
	1023	1.75								
	1273	1.73								
	1523	1.76								
	1773	1.91								
	2023	2.18								
	2273	2.55								
	2523	2.98								
	2773	3.36								

(1) Obtained by regression analysis of all observations carrying equal statistical weight.

$$(2) \sigma = \left| \frac{(\sum \lambda_{\text{obs}} - \sum \lambda_{\text{calc}})^2}{i-p-1} \right|^{1/2}, \text{ where "i" represents the number of data pairs and "p" the degree of the polynomial function.}$$

$$(3) r = \left| \frac{[a_1 \sum (T_i - \bar{T})(\lambda_i - \bar{\lambda}) + a_2 \sum (T_i - \bar{T})^2 (\lambda_i - \bar{\lambda}) + \dots + a_p \sum (T_i - \bar{T})^p (\lambda_i - \bar{\lambda})]}{\sum (\lambda_i - \bar{\lambda})^2} \right|^{1/2}$$

ranged from 9.53 to 25.4 mm; whereas, the RF technique employed paths ranging from 1.55 to 4.75 mm. Accordingly, it should be observed that the CA/GHP-RF interfacial region (1000 - 1300 K) may be described collectively as a macro-micro union which contributes to the λ value biases between methods. The considerable aggregate imprecision of all methods is thought to be attributable, in large part, to gross material nonhomogeneity, as the porosimetric findings (Table 1) connote. Repetitive RF tests were employed in an attempt to segregate, and thus differentiate, between the absolute measurement error and that due primarily to material nonuniformity. The results for LF carbon foam are displayed by the graph of Figure 12 with identical nonlinear regression analyses being applied to four individual (dashed) determinations to generate a composite (solid) regression line. Covariance analyses of the dashed trend lines about the composite (regression) line of Figure 12 were conducted to compute the adjusted 90% tolerance limits, as portrayed in Figure 13, with extrapolation to 273 K included but admittedly invalid below 600 K. On the presumption of a normal distribution of data pairs about the solid regression line of Figure 12, the basis for allocating sources of error between method and material would be sound. However, the dashed trend lines predominantly lie above the computed regression line of Figure 12, which clouds the issue. On the other hand, assuming an abnormal (non-Gaussian) distribution for the observed data and comparing tolerance intervals of Figures 9 and 10 on a quantitative basis with that of Figure 13 leads to the conclusion that material nonhomogeneity and not the method(s) is the major contributor to measurement imprecision.

Stated accuracies⁽¹⁷⁾ of the CA and GHP techniques are, respectively, ± 5 to $\pm 10\%$ and ± 2 to $\pm 5\%$, absolute. For nonhomogeneous materials, these ranges serve only as rough guidelines. All thermal-conductivity results of insulation materials in the range of carbon foam may be viewed as somewhat provisional in the absence of well-characterized standards for comparison and calibration.

Figure 12. ILLUSTRATION OF THE AGGREGATE REPEATABILITY OF THE RADIAL-FLOW TECHNIQUE FOR CARBON FOAM.

Figure 13. THERMAL CONDUCTIVITY OF LOW-FIRED CARBON FOAM WITH AN ADJUSTED 90% TOLERANCE LIMIT.

COMPARISON WITH OTHER FINDINGS

It is oftentimes enlightening and always interesting to compare test results for a given material with similar data exuded from other experimental findings and particularly those hybridized by semitheoretical treatments.

Comparison with the findings of Sandia Laboratories based on both experimental results by the flash diffusivity technique on Y-12-produced 155 and 178 kg/m³ LF material (18,19) and with experimental results from Battelle Memorial Institute's steady-state apparatus for 94 kg/m³ carbon foam, as given in Table 5, shows general trends for the LF material that concur with the mathematical model which is herein proposed (Table 4). Corll's experimental data, zero-time effective conductivity, and computed pore contributions for selected sizes are exhibited in Figure 14 without further explanation. The curves were obtained by highly sophisticated, but plausible, analyses which are fully described in his paper. (19)

CONCLUSIONS AND RECOMMENDATIONS

After several refinements of measurement and data-reduction techniques, particularly with respect to the "nonstandard" radial flow approach, this characterization has yielded what is believed to be carefully acquired, objectively treated values of thermal conductivity as reliable as any which have been, heretofore, reported. Y-12's findings are acknowledged to be provisional and potentially at variance with data obtained by other test techniques. Unfortunately, at high temperatures, all methods are "nonstandard", because none bear the ASTM certification or even general acceptance (due, primarily, to scant confirmatory or

Table 5
THERMAL CONDUCTIVITY RESULTS FOR LOW-FIRED CARBON FOAM

Laboratory (1)	Method (2)	Source of Material	Material Density (kg/m ³)	Direction of Measurement	Temperature (3) (K)	Thermal Conductivity (3) (watt/m-K)
Sandia	Flash Diffusivity	Bendix - Kansas City	94	Parallel to Direction of Cell Rise	1173	0.4
					1573	0.6
					1873	0.8
					2173	1.2
					2473	1.4
					2573	2.4
Battelle Memorial Institute	Steady-State Longitudinal Heat Flow Apparatus	Bendix - Kansas City	94	Parallel to Direction of Cell Rise	1173	0.5
					1573	0.6
					1873	0.9
					2173	1.3
					2473	1.0
					2573	3.4
Sandia	Flash Diffusivity	Bendix - Kansas City	94	Perpendicular to Direction of Cell Rise	1273	0.2
					1573	0.3
					1873	0.5
					2173	0.7
					2373	0.8
					2473	1.0
Battelle Memorial Institute	Steady-State Longitudinal Heat Flow Apparatus	Bendix - Kansas City	94	Perpendicular to Direction of Cell Rise	2573	1.1
					1273	0.3
					1573	0.4
					1873	0.7
					2173	0.9
					2373	1.2
Sandia	Flash Diffusivity	Oak Ridge Y-12 Plant	155	Undefined	2473	1.4
					2573	2.1
					773	0.5
					1023	0.8
					1273	1.0
					1523	1.3
Sandia	Flash Diffusivity	Oak Ridge Y-12 Plant	178	Undefined	1673	1.4
					773	0.3
					1023	0.4
					1273	0.5
					1523	0.8
					1623	1.0

(1) These data are reported by Rainhart(18) and Corli.(19)

(2) Test environments not disclosed, but presumably were vacuum.

(3) Values obtained from graphical display and are, therefore, approximate.

Figure 14. COMPARISON OF BATTELLE EXPERIMENTAL MEASUREMENTS AND SANDIA THEORETICAL CALCULATIONS FOR 94 kg/m³ LOW-FIRED CARBON FOAM (AFTER CORLL(19)).

comparative analyses of similar materials), and because there is a lack of accepted standard reference materials. Until such time that carbon foam and other similar types of insulative materials are examined by a number of careful experimenters using diverse techniques and thorough documentation, questions about test accuracy, applicability, and reliability will prevail.

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