THERMAL DIFFUSIVITY OF SX-5 GRAPHITE FROM 800° TO 2800°C

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ABSTRACT - The thermal diffusivity of SX-5 graphite in orthogonal axes was measured from 800° to 2800°C by the laser flash technique in a vacuum and in a helium atmosphere. A neodymium glass laser system is the pulsed energy source; a photomultiplier tube detects the transient-time temperature response of the thin samples. Cowan's method for correction of the heat loss and a finite pulse time correction are included in the thermal diffusivity calculations. The thermal diffusivity data are fitted to a function in reciprocal temperature by a least squares technique. The precision of the flash technique is certified by repeated measurements on many samples. The accuracy of the measurements is estimated by comparison with published high temperature thermal conductivity data on other graphites and on Armco iron.

KEY WORDS - Thermal diffusivity, thermal expansion, heat capacity, thermal conductivity, Armco iron, SX-5 graphite, AAQ graphite, laser flash, equations for thermal diffusivity.

I. INTRODUCTION

Graphite is utilized in the advanced nuclear rocket motors developed at the Los Alamos Scientific Laboratory because it has excellent mechanical and thermo-physical properties at very high temperatures. Calculation of thermal stresses and heat transfer rates requires accurate values for the thermal conductivity or the related parameters, thermal diffusivity, thermal expansion, and heat capacity. Furthermore, since graphite is usually anisotropic, variations in these parameters with respect to structural orientation should be delineated.

This paper describes the measurement of the thermal diffusivity in the orthogonal axes of an extruded, fine-grain graphite[2], SX-5, from 800° to 2800°C. The experimental precision was enhanced by smoothing the data from scores of measurements by a least squares technique. The accuracy of the computed values is estimated by comparison with published thermal conductivity data (via the relation that the thermal conductivity is the product of the thermal diffusivity and the specific heat per unit volume). In addition, the laser flash apparatus is standardized by comparison of its thermal diffusivity measurements on a "round robin" sample of Armco iron with other published values.

The pulse method as proposed by Parker et al [1][3] has been extensively developed in this decade to measure the thermal diffusivity of small samples rapidly and precisely.

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[1] Staff Member.
[2] Manufactured by Speer Carbon Company.
[3] Numbers in brackets designate references listed at the end of this paper.

DISCLAIMER

Essentially, the method consists of applying an instantaneous heat pulse to one face of a thin disc and measuring the time-temperature response of the opposite face. The heat flow through the sample is described by the general Fourier equation for cylindrical coordinates with no internal heat generation:

$$\Delta^2 T\ (x,r,t) = \alpha^{-1} \frac{\partial T(x,r,t)}{\partial t} \tag{1}$$

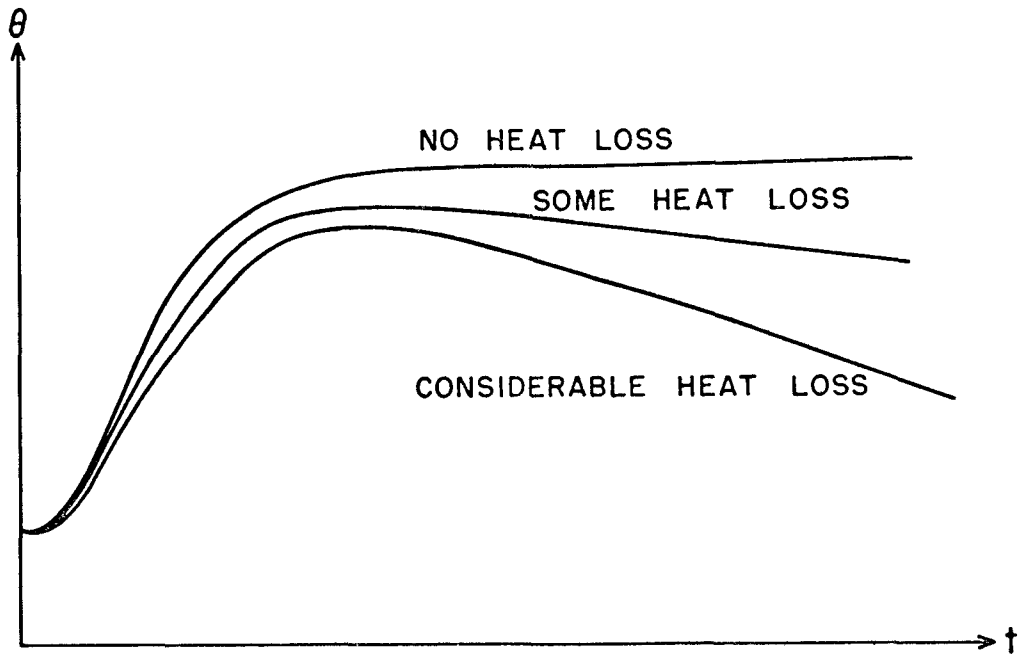
Several simplifying assumptions were made by Parker in the analytical solution of the general equation. These include 1) one dimensional heat flow, 2) the heat pulse absorbed instantaneously in an infinitesimally thin layer on the front of the sample, and 3) no heat losses by conduction or radiation from the faces of the sample. These assumptions lead to a simple solution of equation (1):

$$\alpha = \frac{0.139\ell^2}{t_{1/2}} \tag{2}$$

where α is the thermal diffusivity (cm^2/sec), ℓ is the sample thickness (cm), and $t_{1/2}$ is the time required for the back face temperature to attain one-half its maximum value (sec).

Equation (2) has been used to calculate the thermal diffusivity of Armco iron by G. L. Denman [2], R. E. Taylor [3], and R. A. diNovi [4], the thermal diffusivity of ZTA and SX-5 graphite by P. Wagner [5, 6], and the thermal diffusivity of BeO by R. J. Freeman [7].

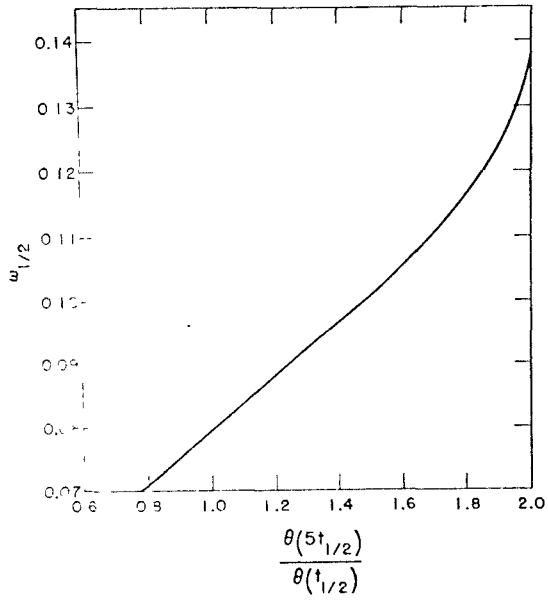
Cape and Lehman [8] solved the general heat equation for heat loss from the sample and predicted temperature vs. time plots of these general shapes:



Now the thermal diffusivity is given by:

$$\alpha = \frac{w_{1/2}\ell^2}{t_{1/2}} \tag{3}$$

where the constant in eq. (2) has been replaced by the parameter, $w_{1/2}$, which is a function of the heat loss. This parameter is determined from a graph given by Cowan [9]:



Experiments on Armco iron sleeves [10] demonstrated that the $w_{1/2}$ term accounts for edge losses from a disc as well as radiation losses from the faces of the disc.

Cape and Lehman [8] also derived a complex solution of the heat equation which is not restricted by requirement of instantaneous absorption of the heat pulse on the front face of the sample. A more straightforward approach used in this work consists of measuring the rise time to maximum laser pulse intensity when the capacitor bank is discharged at different levels. This time is subtracted from $t_{1/2}$ as defined in eq. (2). The numerical value of this correction varies from 0.4 to 0.8 milliseconds which usually meets Cape and Lehman's criterion of $t_c > 50\ T$ for 1% accuracy (t_c is the characteristic rise time of the back surface and T is the pulse duration).

The accuracy of the thermal diffusivity measurements on SX-5 graphite could not be determined because no other measurements have been published. However, samples of another type of graphite, AAQ-43, whose thermal conductivity had been measured by P. Wagner [12], were available for thermal diffusivity measurements. Thermal conductivity and thermal diffusivity can be compared by the equation:

$$\lambda = C_p d\alpha \qquad (4)$$

where λ is the thermal conductivity in $W\ m^{-1}\ C^{-1}$,
C_p is the heat capacity in $J\ kg^{-1}\ C^{-1}$,
d is the density in $Kg\ m^{-3}$, and
α is the thermal diffusivity in $M^2 s^{-1}$.

Values for the density can be determined from thermal expansion data given in Wagner's paper [12]. The heat capacity data on this particular graphite are not available so literature values for graphite must be used. However, the author of a generally accepted compilation [11], G. B. Spence, states, "Except near absolute zero, the Cp's of all types of natural and specific graphites are essentially the same and the differences between the

values at high temperatures are less than the experimental error." Therefore, reasonable comparisons between thermal conductivity and thermal diffusivity of graphites may be made by equation (4).

II. EXPERIMENTAL PROCEDURES AND APPARATUS

1. Sample Characterization

The SX-5 graphite was received from the manufacturer in the form of large rectangular slabs as sketched in Figure 1. The extrusion direction is designated as the X axis and the Y and Z axes are at right angles to it. The thermal diffusivity discs were machined from 1" x 1" x 4" bars which were cut from the approximate center of a slab. The samples for thermal expansion and electrical resistivity measurements were taken from adjacent portions of the same slab. Discs adjacent to those used for diffusivity tests were examined for porosity in the three orientations at a 50X magnification. The photomicrographs[4] in Figure 2 reveal a structure with considerable large porosity (the dark areas) and little orientation. P. Wagner [6] reports that this porosity is larger than usual in other graphites and the pores are open to both Hg and He intrusion.

The manufacturer describes SX-5 as a fine-grain, extruded graphite which is fabricated from calcined coke and coal tar pitch and graphitized at a maximum temperature of 2800°C. Some previous measurements at this laboratory had indicated dimensional changes in SX-5 at high temperatures. Therefore, a series of preliminary thermal diffusivity tests was run to confirm the material stability. The data in Table 1 show that the thermal diffusivity was constant for 185 minutes at 2530°C. Even an additional 30 minutes at 2790°C decreased the diffusivity only the expected amount. Since routine thermal diffusivity tests submitted

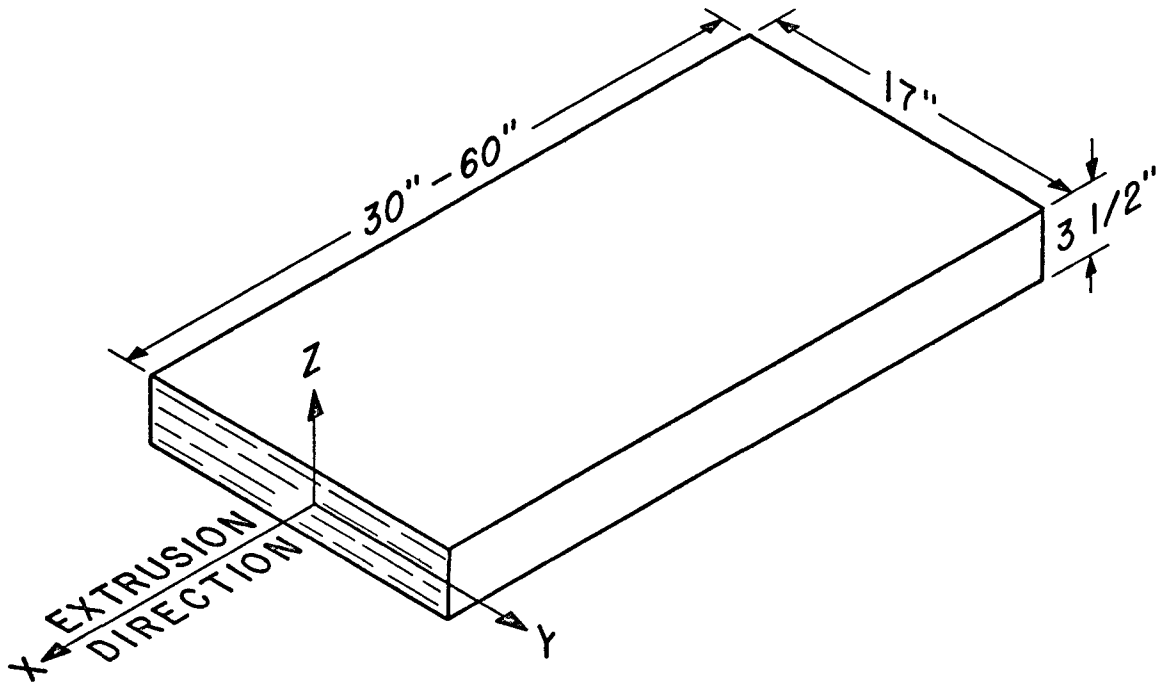
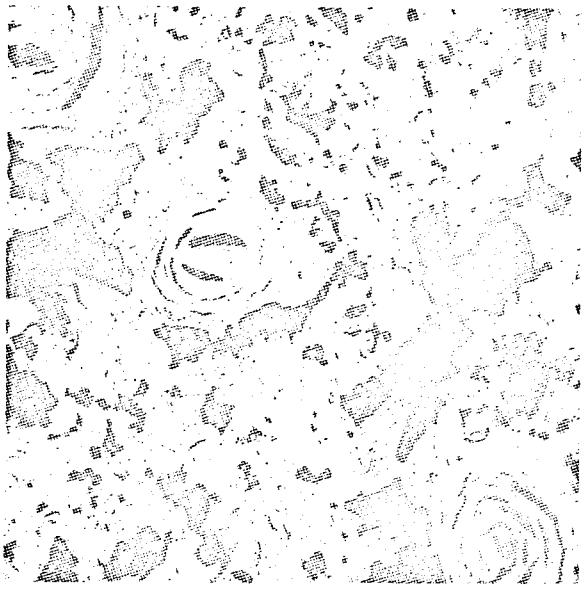
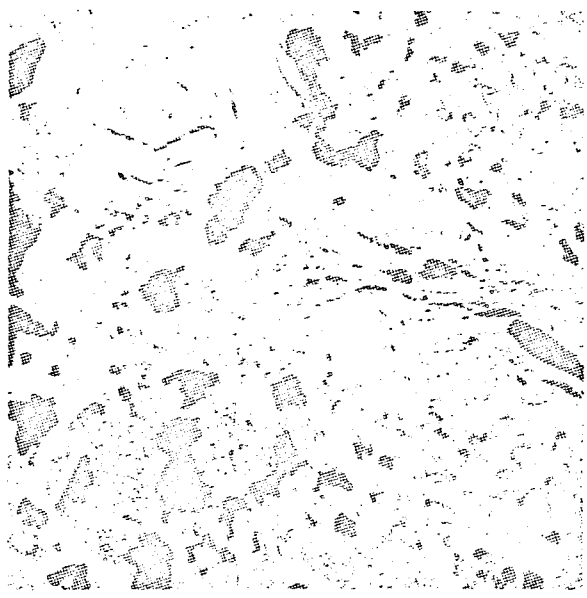


Figure 1. Orthogonal Axes in SX-5 Graphite

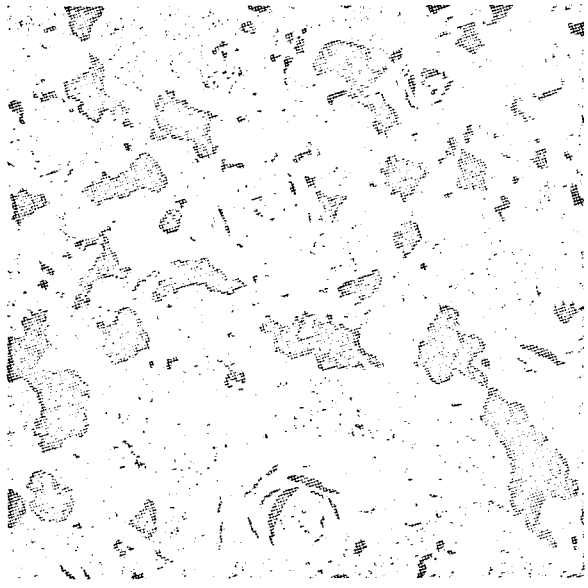
[4] Courtesy of C. G. Hoffman, this laboratory.



X Axis, Vertical



Y Axis, Vertical



Z Axis, Vertical

Figure 2. Photomicrographs of SX-5 Graphite, 50X

Table 1. Temperature-Time Effects on Thermal Diffusivity of SX-5 Graphite

Temperature °C	Time at Temperature minutes	Thermal Diffusivity M x 10^{-4} sec^{-1}
2530	5	0.086
2530	35	.084
2530	185	.085
2790	30 (a)	.083

(a) Preceded by 185 minutes at 2530°C

Table 2. Characterization of SX-5 Graphite

	Axial Orientation		
	X	Y	Z
Electrical Resistivity, Ω m x 10^{-6}	9.81 ± .03	9.43 ± .03	11.14 ± .14
Coefficient of Thermal Expansion, m/m°C	7.05 x 10^{-6}	7.20 x 10^{-6}	7.56 x 10^{-6}
Density, Kg m^{-3} x 10^{3}	1.66	1.68	1.67
% Density Decrease after 15 Min. at 2750°C	0.4	0.2	0.2

the samples to the extreme temperatures for only 15 minutes, the diffusivity was confirmed as independent of time at temperature during the test period.

The electrical resistivity was measured at room temperature by the four probe method with four determinations on each of four 1" x 1" x 4" bars from each axis. Bulk density determinations were made by weighing and measuring the thermal diffusivity discs. The coefficients of thermal expansion were calculated from equations generated as discussed under thermal expansion later in this report. These data are collected in Table 2.

The sample of "round robin" Armco iron used for standardizing the flash diffusivity apparatus came from the same reference bar from which samples have been distributed to 15

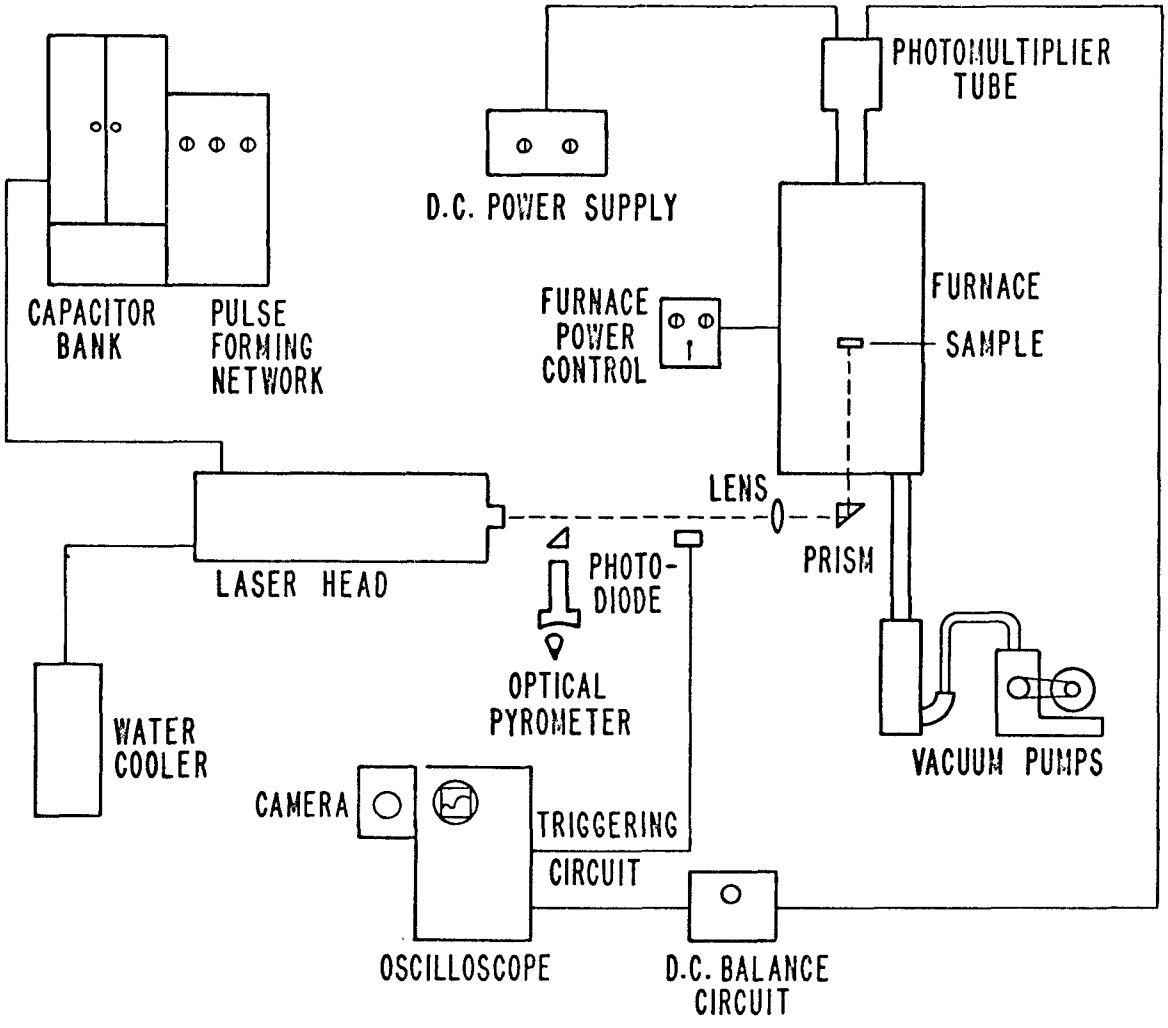


Figure 3. Schematic of Thermal Diffusivity Apparatus

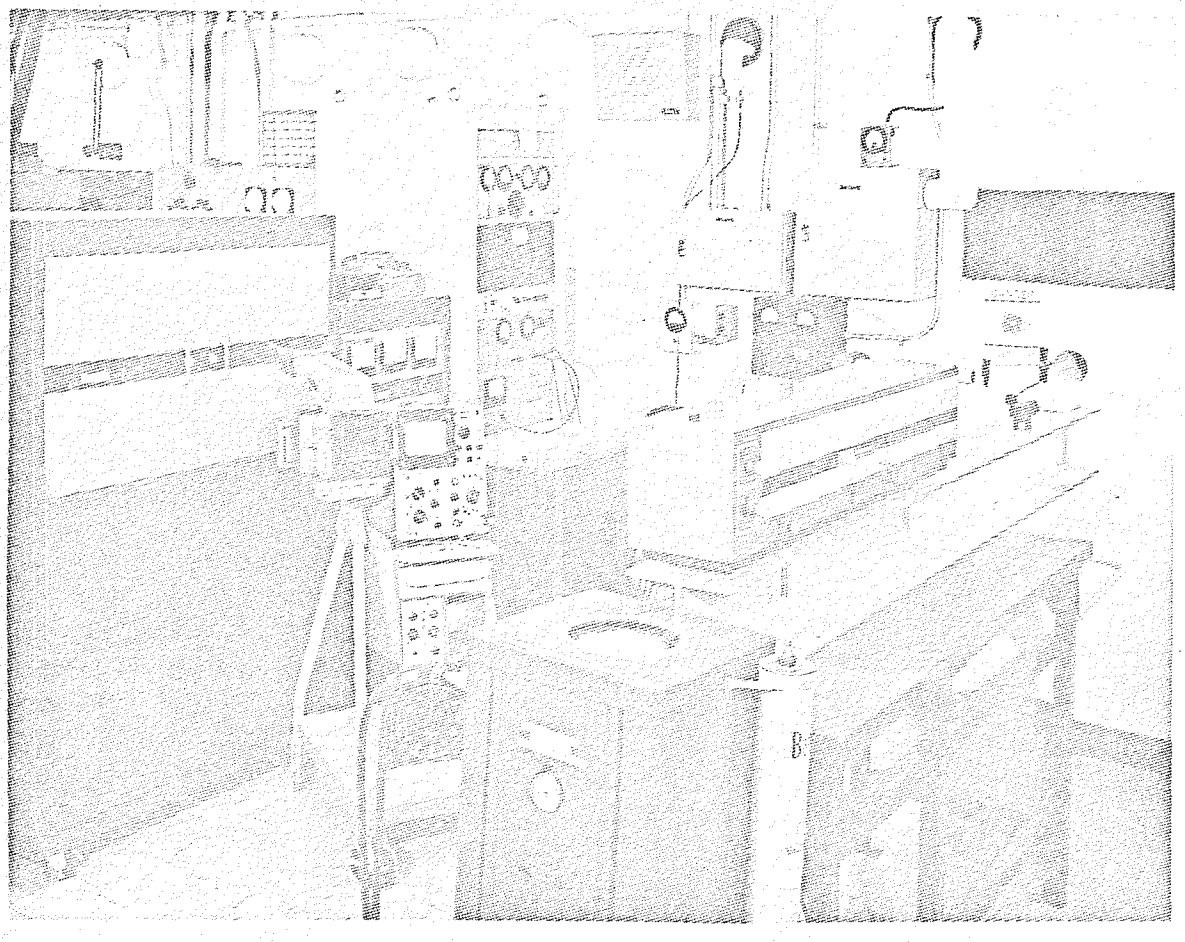


Figure 4. Laser Head, Pulse Forming Network, and Recording System

laboratories active in thermal measurements. The sample of AAQ graphite used for high temperature comparison was cut from the same stock that Wagner [12] used. This graphite is a polyfurfuryl alcohol bonded material which has been graphitized at 2800°C, has a density of 1.901 g/cm^3, and has an electrical resistivity (parallel to the extrusion direction) of 11.60 x 10^{-6} Ω m.

2. Laser Thermal Diffusivity Apparatus

The schematic (Fig. 3) and the photographs (Figs. 4 and 5) illustrate the thermal diffusivity apparatus.

The 100 joule laser system (TRG Model 302) consists of an 8 kilojoule power supply, a pulse forming network, and a laser head containing two identical, series cycled units each having a flash lamp and crystal. The laser cavities are supplied with interchangeable ruby (0.05% Cr) and glass (1-3% Nd) rods for emission of electromagnetic radiation at 0.694 and 1.06 microns respectively; the Nd rods were used exclusively in this work. The rods and flash tubes are cooled by chilled, deionized water circulating through the laser heads. The charge level of the capacitor bank is continuously preselectable from one to eight kilojoules; four or six kilojoules were used on the SX-5 graphite depending on the sample thickness and temperature.

The vertical high temperature furnace (Fig. 5) has interchangeable heating elements for

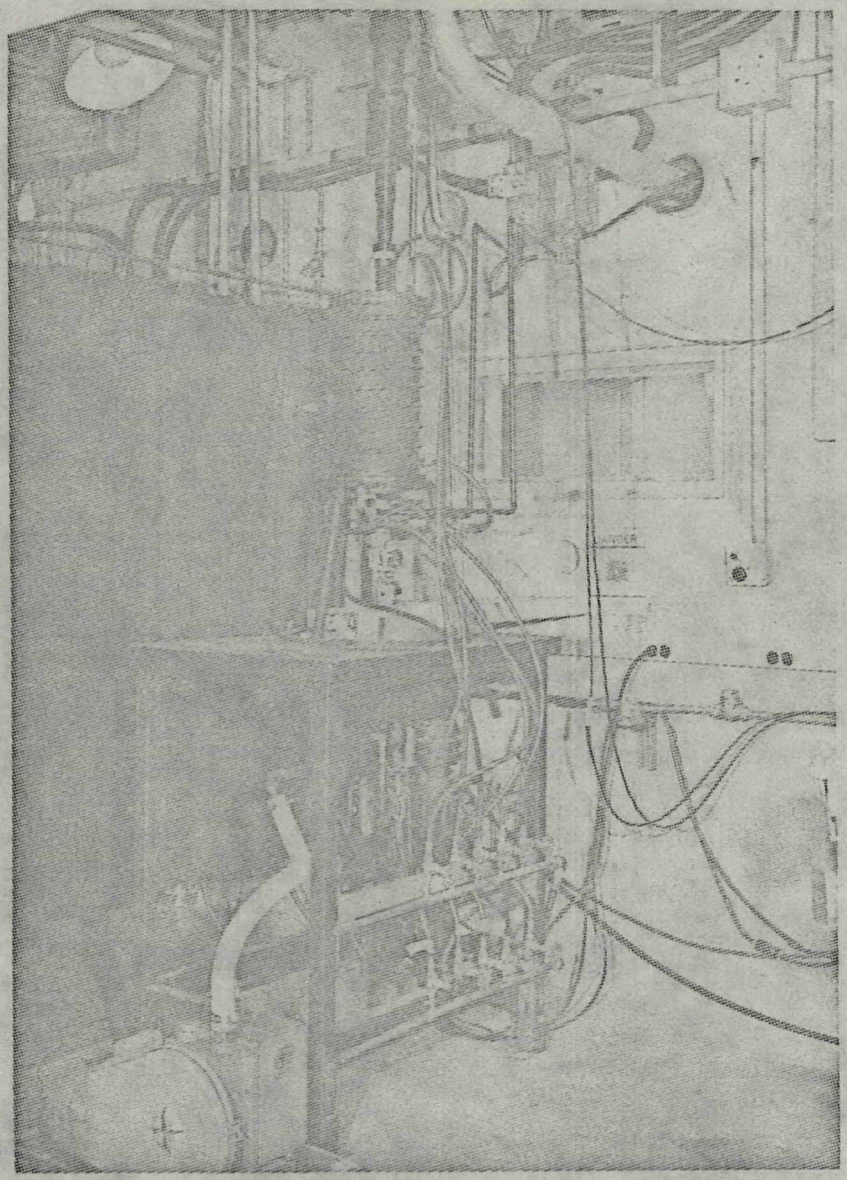


Figure 5. Sample Furnace, Photomultiplier Tube and Vacuum System

high and low temperature work, i.e., a 0.015 inch wall Ta tube wrapped with 3 mil Ta sheet for measurements to 2000°C and a 1/16 inch wall graphite tube wrapped with carbon felt for heating to 2800°C. A motor generator with variable field excitation supplies DC to the heating element. The DC represents a significant improvement over the AC used previously [13] because it eliminates the 60 cycle continuum which had been superimposed on the cathode ray tube display. The furnace vacuum system, a M-1402 Duo-Seal forepump and a 4 inch CVC oil diffusion pump, can evacuate the system to 1×10^{-5} torr in five minutes. The furnace can be filled with an inert gas to 3 psig. This inerting is mandatory above 2200°C because the heating elements deteriorate rapidly in a vacuum at very high temperatures. The furnace windows are of 1/8 inch quartz and the top of the furnace is hinged for easy access to the sample holder assembly.

The one-half inch diameter sample discs which may be from 0.020 to 0.100 inches (0.05 to 0.254 cm) thick are supported in a stainless steel tube for low temperature measurements or a carbon tube for high temperature observations (Fig. 6). The stainless steel holder was utilized for the calibration measurements on the Armco iron sample from 25° to 1000°C;

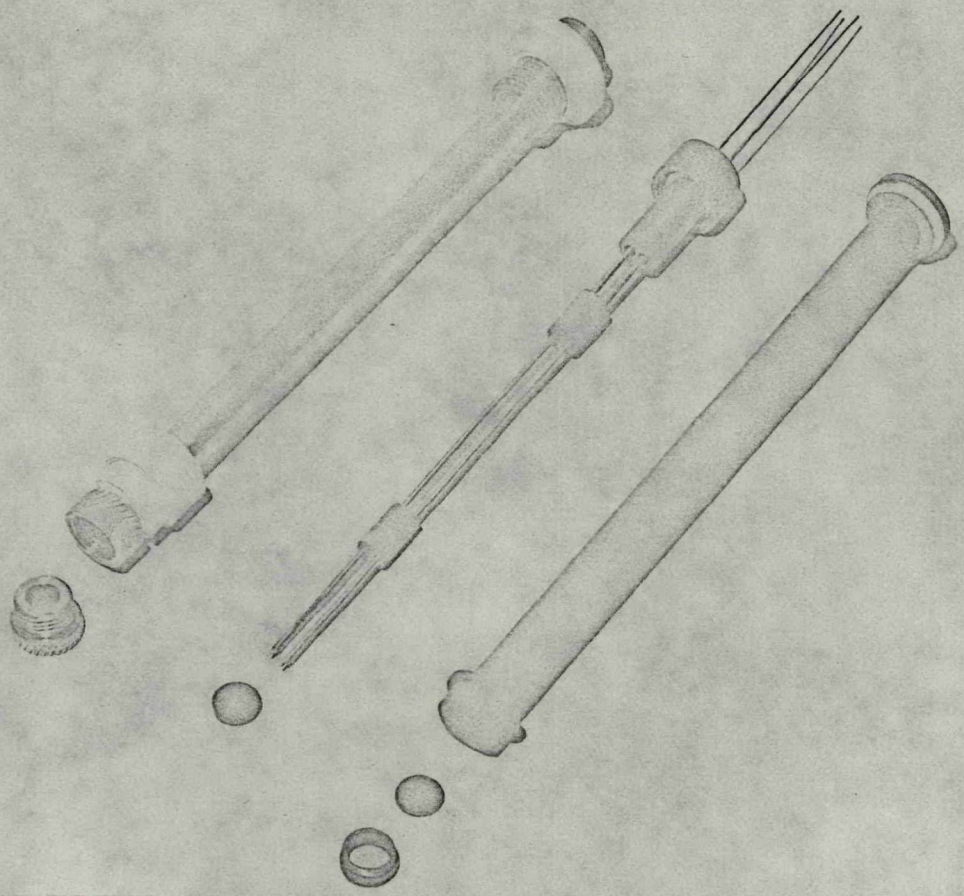


Figure 6. Stainless Steel and Carbon Sample Holders

the carbon model was used for the graphite determinations.

The low range (25° to 1000°C) temperature response of the samples after fulguration is measured by pressing sharpened, 20 gauge chromel and alumel thermocouple wires against the back of the disc and recording their electrical output. The alumina insulated thermocouple wires are supported by spring-loaded, stainless steel sleeves. Since the points of the thermocouple wires are separated by 3/16 of an inch, the actual electrical junction is through the test material. The procedure eliminates thermal contact problems which are probable when a thermocouple bead is pressed against the sample.

The microvolt thermocouple output is conducted via shielded cable to a Tektronix 2A61 differential amplifier and displayed on a long persistence Tektronix 564 oscilloscope equipped with a 3B3 time base unit. The heat pulse displays are photographed on type 46L positive transparency film with a Polaroid Land Camera (Model C-12). The transparency is projected onto 10 x 10 cm grid paper from the requisite range to overlay the paper grid with the image of the cathode ray tube grid. The heat pulse image is traced on the paper and the $t_{1/2}$ and $\theta(5t_{1/2})/\theta(t_{1/2})$ data are recorded. This enlarging technique enhances the data quality because the original films are too small for precise measurements. The sample temperature before fulguration is measured by a potentiometer using the sharpened thermocouple probes and a reference junction.

High temperature thermal diffusivity measurements are made on the graphite discs supported by the carbon tube (Fig. 6) which is centered in the vertical heater tube by means

of a boron nitride collar. After the front face of the sample is heated by the laser pulse, the temperature response of the opposite face is monitored by a RCA-6655A photomultiplier tube mounted on top of the furnace (Fig. 5). An iris and filter are positioned in the light path before this tube to attenuate the furnace incandescence at temperatures above 2000°C. A Power Designs Pacific Co., Model HV-1565, regulated DC power supply energizes the phototube anode.

The amplified signal from the photomultiplier tube is divided for improvement of the tube operation at high background light levels. A portion of the signal flows through a one megohm resister to a positive power supply (Barnes Corporation Model 32-101). This positive voltage is adjusted to reduce the anode voltage to ground potential corresponding to the background light being emitted from the furnace at that particular temperature. The modified photomultiplier tube output is displayed and photographed on the cathode ray tube screen as previously described.

The laser head and a moveable prism are mounted on a 6 foot long aluminum I beam. The prism can be raised into the light path so that a Leeds and Northrup optical pyrometer is aligned with the front face of the sample. In usual practice, the sample temperature is measured optically, the prism is lowered, and the laser pulse is fired against the sample. The sample temperature remains constant within the precision of the optical measurement for the 10 to 20 seconds required for the above procedures. A second optical bench, separated from the first by a 6 inch thick concrete blast wall, supports a 30 cm. focal length lens, a prism, and the vertical tube furnace. The blast wall, a precaution for future work with hydrogen atmospheres, is perforated by a 4 inch diameter hole for passage of the laser beam. The lens focuses the laser beam to a 5/16 inch diameter spot on the sample and the prism deflects the horizontal beam up into the vertical furnace. Exact impingement of the entire laser pulse on the samples is insured by 1) test pulses on dummy samples covered with black paper which is scorched by the laser pulse and 2) water-cooled electrode holders which prevent any movement of the heater tube or sample holder by thermal expansion of the furnace components. Precise alignment of the laser beam with the sample is the most difficult technical detail of this experiment. This difficulty was resolved by fabrication of a precision prism holder which permitted very small, reproducible adjustments of the prism in three planes.

The oscilloscope sweep trace is triggered by a 1.5 volt signal from a SD-100 photodiode which intercepts a portion of the flash tube light as it pumps the laser rods. This diode is a silicon surface barrier device containing a p-n type junction on the semi-conductor surface. Triggering by this method is very reliable since it does not depend on any circuitry within the pulse forming network of the laser. Thus, the initial point of the heat pulse is reproducible and corrections for the finite pulse time can be calculated from a rapid (0.5 millisecond per centimeter) sweep rate display of the laser pulse itself.

The Leeds and Northrup optical pyrometer was calibrated against a NBS standard tungsten filament lamp. Corrections were also made for the quartz window, lens, and prism light absorption. The emissivity of the graphite samples was determined as unity by drilling black body holes into a thick sample and comparing the interior temperature to the surface temperature -- no difference was observed.

3. Thermal Expansion Apparatus

The linear thermal expansion of SX-5 graphite in the orthogonal axes was measured in a 120 inch long, 6 inch diameter, graphite tube resistance furnace. In a typical measurement, a graphite cage containing a 30 inch stack of graphite specimens is centered in the heater tube and the entire assembly is heated at a rate of 3° to 4°C per minute to 2500°C. The sample temperature is determined with a Pt-Pt 10 Rh thermocouple from 20° to 800°C and with an optical pyrometer over the remaining range. The linear thermal expansion is measured with twin 5X telemicroscopes mounted on a horizontal optical bench. These telemicroscopes are sighted on pointed graphite fiducials which are embedded in specimen segments 29 inches apart. Incandescent lamps in viewports on opposite sides of the furnace backlight

the fiducials until their temperature is sufficient for self-illumination. One atmosphere of helium is maintained in the furnace. Under these conditons, the algebraic sum of the telemicroscope carriage movements corresponds to the net specimen expansion. An IBM 7094 computer program determines a least squares fit of the measured linear thermal expansion versus temperature. Constant terms of the following equation are derived by the program:

$$\frac{\Delta L}{L_o} = A(T-20) + B(T-20)^2 + C(T-20)^3 + D(T-20)^4 \qquad (5)$$

where ΔL is the cumulative expansion to T, the centigrade temperature, and L_o is the initial gauge length at 20°C.

III. RESULTS

1. Thermal Diffusivity of Armco Iron Standard

Sixty-five thermal diffusivity determinations of Armco iron were made over the 14° to 1100°C temperature range. The 14° to 782°C data were submitted to a computer program composed by R. H. Moore and R. K. Zeigler [14] which fitted them to this equation in reciprocal temperature:

$$\alpha = -0.1644 + \frac{292.482}{T} - \frac{9.316 \times 10^4}{T^2} + \frac{1.136 \times 10^7}{T^3} \qquad (6)$$

where α is the thermal diffusivity in cm^2/sec and T is the absolute temperature in degrees K. The standard deviation of the predicted mean value of α in eq. (6) varied from 0.0010 to 0.0018 (± 5% at the maximum temperature) over the 14° to 782°C interval. Least squares fits were computed for the cases which omitted the (T^{-3}) and $(T^{-2} + T^{-3})$ terms. However, these curves did not fit the observed data satisfactorily so eq. (6) was selected.

The computed data from the equation are sketched on Figure 7 from 14° to 782°C; individual points are marked at the higher temperatures above the α-γ transition point. Data points from papers by DiNovi [4] and Freeman [7] are also shown on Figure 7. The agreement among the three sets of data is about ± 3%. Since our measured precision is ± 5%, our apparatus and technique are standardized and therefore qualified for thermal diffusivity measurements on unknown materials at least over the low temperature range. By induction, we extrapolate this qualification to higher temperatures despite the lack of certified high temperature standards.

2. Thermal Diffusivity of SX-5 Graphite

Most reported thermal property measurements have been made in a vacuum to eliminate convection effects from the conductive gas in contact with the sample. This is manifestly impossible above 2200°C because of the high vapor pressure of carbon and consequent deterioration of the heating element. Therefore, thermal diffusivity measurements were done in a vacuum from 800° to 2200°C and again in helium from 800° to 2800°C. Specimens from the X axis of graphite slab number 1 were tested alternately under both conditions to nullify any effects due to sample constitution. Forty-six data points in vacuum and 34 in helium were submitted to the least squares computer program and fitted to three equations in 1/T, i.e., $\alpha = A + B/T$, $\alpha = A + B/T + C/T^2$, and $\alpha = A + B/T + C/T^2 + D/T^3$, where T is now the temperature in °K.

A preliminary examination of the data fits suggested that the first equation ($\alpha = A + B/T$) was adequate to describe the thermal diffusivity-temperature relationship. The hypothesis was verified by applying the "f" test from Wm. Volk [15]. The first degree equations

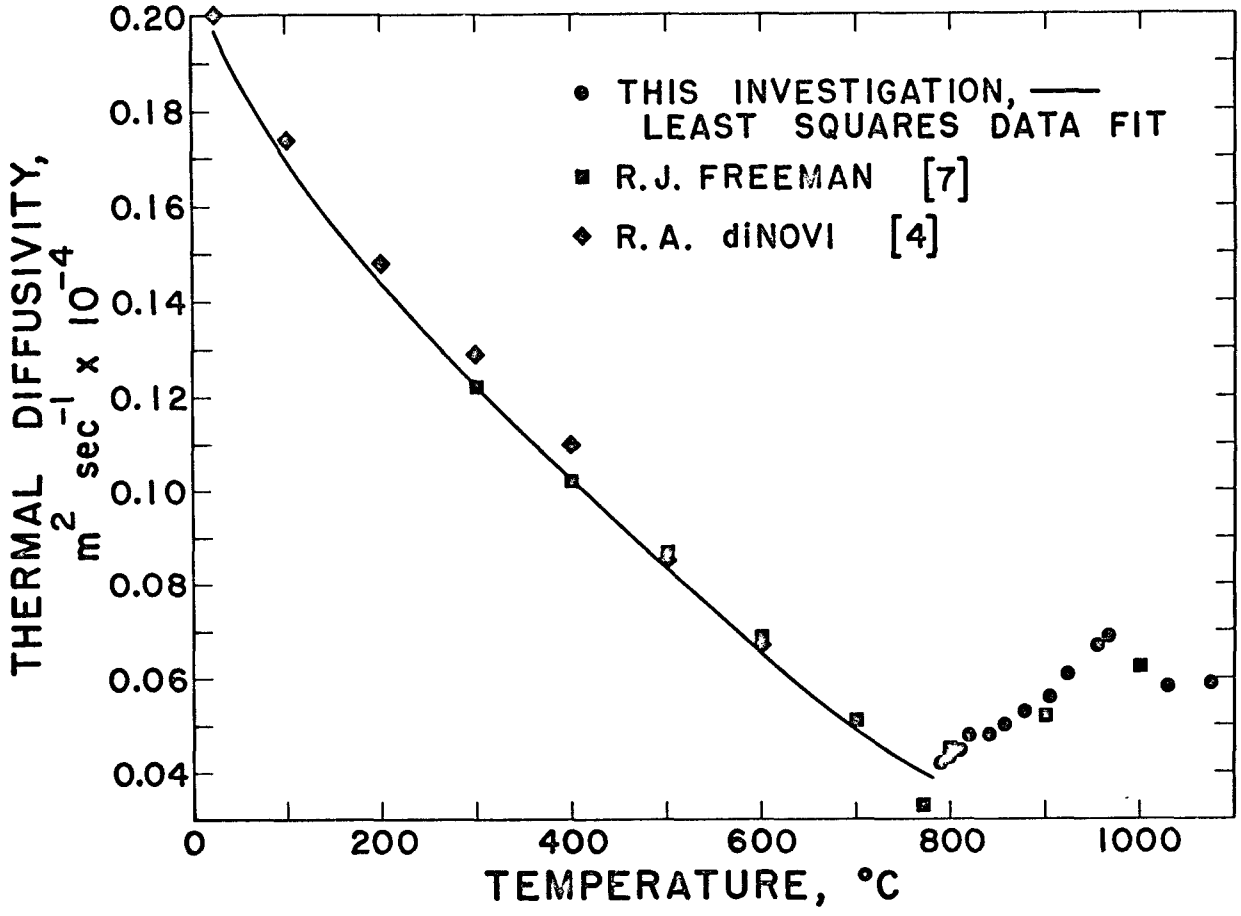


Figure 7. Thermal Diffusivity vs. Temperature of Armco Iron

for the least squares fits in vacuum and helium are compiled in Table 3 and are plotted in Figure 8.

Table 3. Equations for the Thermal Diffusivity of SX-5 Graphite in Vacuum and in Helium

Slab Number 1, X axis	Data Points	$\alpha = A + B/T$ (1073 - 2473°K) (a)			
		A	σ (b)	B	σ (b)
Vacuum (1×10^{-5} torr)	46	0.0385	± 0.0034	148.504	± 5.022
Helium (1 atmosphere)	34	.0319	± .0028	159.310	± 4.888

(a) Maximum T of 3033°K in He
(b) Standard Deviation

Even a cursory examination of the two curves in Figure 8 indicates that the thermal diffusivities as measured in vacuum and helium are the same. A more sophisticated statistical test as given by A. Hald [16] determines that neither the slopes nor the intercepts are significantly different. Therefore, the curves are statistically identical within the 1% confidence level and there is actually no difference in thermal diffusivities as measured

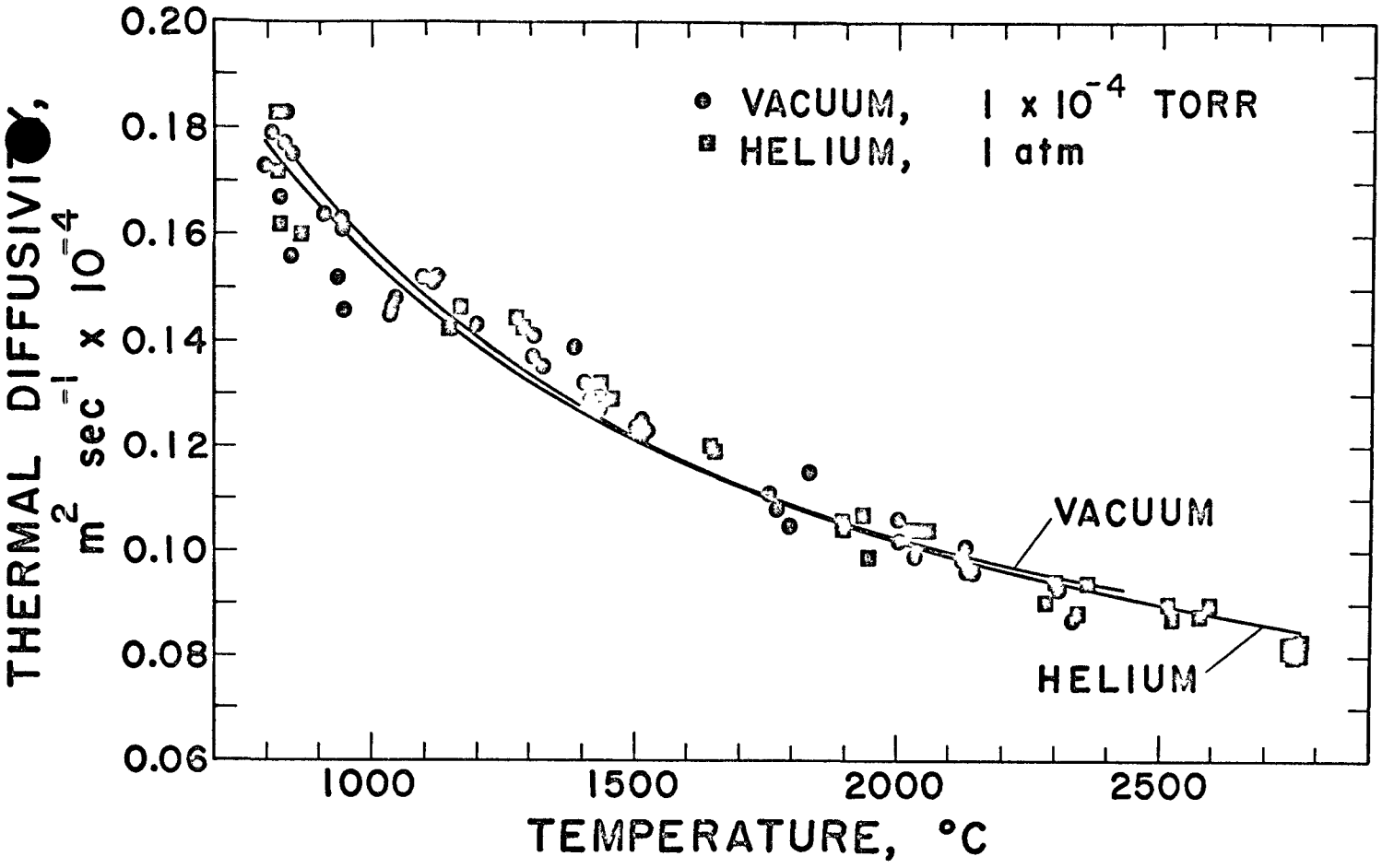


Figure 8. Thermal Diffusivity vs. Temperature of SX-5 Graphite, X-Axis, in Vacuum and in Helium

in a vacuum or in helium. Because of this conclusion, subsequent measurements were made in either vacuum or helium as imposed by experimental convenience.

The second set of thermal diffusivity measurements examined whether there is a difference between slabs of SX-5 graphite. Several "X" samples from each of two slabs were tested and the thermal diffusivity values were submitted to the computer program and statistical analysis. The first degree equations for the least squares fits are listed in Table 4 and plotted on Figure 9.

In this case, a superficial examination of the equations and the curves does not indicate if the two slabs of SX-5 graphite really differ in thermal diffusivity. However, the

Table 4. Equations for Thermal Diffusivity of SX-5 Graphite from Different Slabs

Combined Vacuum and Helium Data, X Orientation	Number of Data Points	$\alpha = A + B/T$ (1073° - 3073°K)			
		A	σ (a)	B	σ (a)
Slab No. 1	80	0.0347	± 0.0021	154.281	± 3.361
Slab No. 2	84	.0405	± 0.0027	139.125	± 4.364

(a) Standard Deviation.

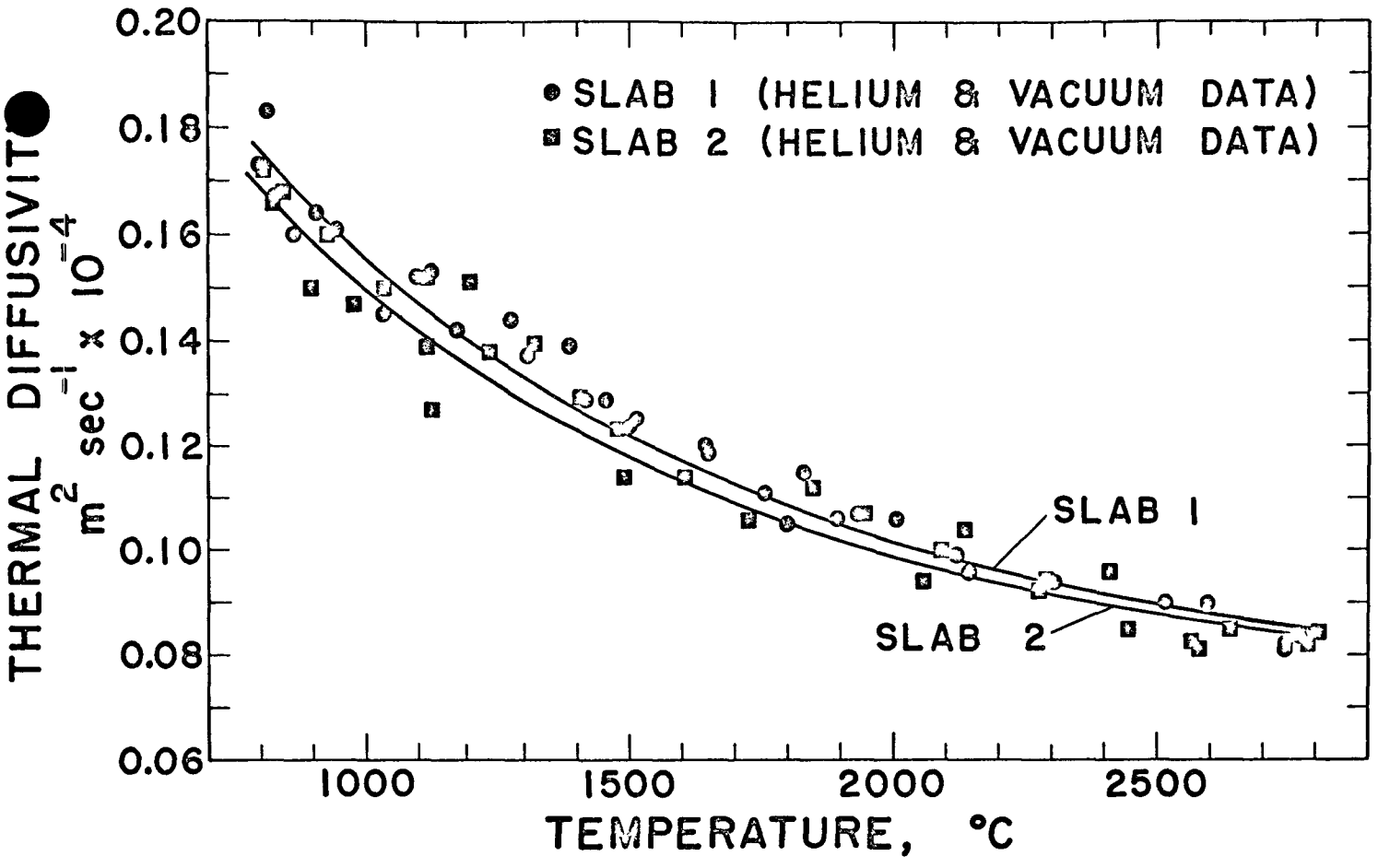


Figure 9. Thermal Diffusivity vs. Temperature of SX-5 Graphite, X Axis, from Different Slabs

same statistical test from Hald [16] determines that the curves are significantly different at the 1% confidence level. Therefore, one may conclude that thermal diffusivity measurements detect differences in the composition or manufacturing history of these graphite slabs.

The third set of measurements compares the thermal diffusivities in the orthogonal axes of specimens from one slab of SX-5 graphite. Two or more samples from each axis were tested in both vacuum and in helium; the data were analyzed by the same computer program and statistical technique. The first degree equations for the least squares fits are listed in Table 5 and plotted on Figure 10.

Table 5. Equations for Thermal Diffusivity of SX-5 Graphite in Orthogonal Axes

Combined Vacuum and Helium Data, Slab No. 2	Number of Data Points	$\alpha = A + B/T$ (1073° - 3073°K)			
		A	σ (a)	B	σ (a)
X axis	84	0.0405	± 0.0027	139.125	± 4.364
Y axis	54	.0383	± .0027	142.089	± 4.802
Z axis	55	.0224	± .0034	162.008	± 5.783

(a) Standard Deviation

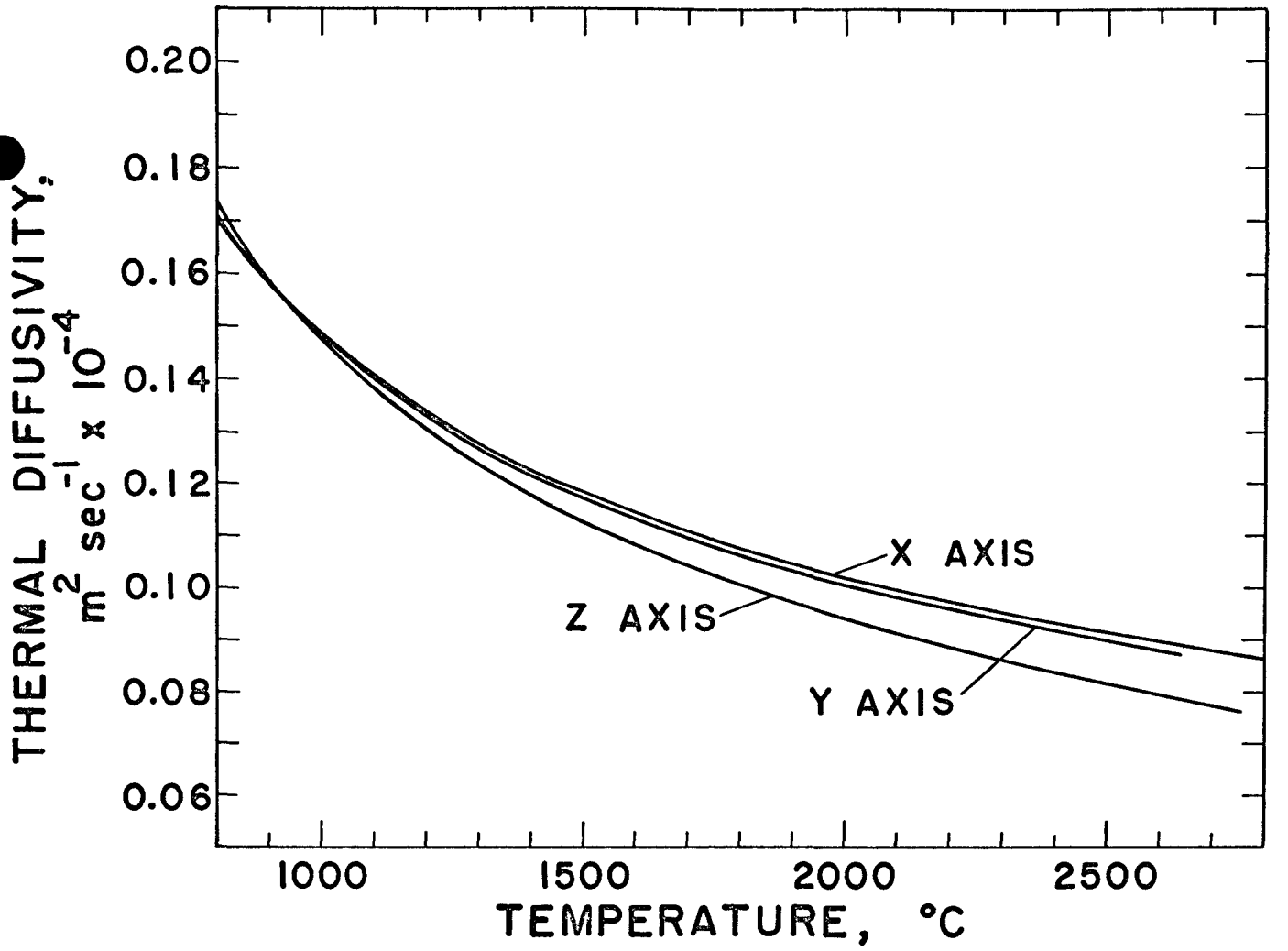


Figure 10. Thermal Diffusivity vs. Temperature of SX-5 Graphite in Orthogonal Axes

A visual examination of the curves in Figure 10 and the parameters in Table 5 implies that the thermal diffusivity in the Z axis may be different from that in the X and Y axes which are apparently indentical. The statistical test from Hald [16] concludes that this is indeed the case, i.e., the X and Y thermal diffusivities are not significantly different at the 1% confidence level but the Z axis thermal diffusivity is significantly different from them.

3. Thermal Expansion of SX-5 Graphite

The observed linear expansion vs. temperature data of SX-5 graphite were fitted to a fourth degree equation in (T-25) where T is the observed temperature in °C. Higher and lower degree equations were tested but the fourth degree expression was the most satisfactory. The analytical expressions for the thermal expansions are listed in Table 6; the percent linear expansion vs. temperature curves are graphed on Figure 11.

Although a statistical analysis was not computed for the thermal expansion curves, a simple "eyeball" test shows that the Z axis expansion curve is again different from the X and Y curves. This is further evidence of the thermal anisotropy of SX-5 graphite.

Table 6. Equations for the Linear Thermal Expansion of SX-5 Graphite in Orthogonal Axes

Slab No. 1	Number of Data Points	$\beta^* = A(T-20) + B(T-20)^2 + C(T-20)^3 + D(T-20)^4$			
		A	B	C	D
X axis	44	4.30×10^{-6}	3.02×10^{-9}	-1.40×10^{-12}	2.62×10^{-16}
Y axis	23	3.88×10^{-6}	4.10×10^{-9}	-1.95×10^{-12}	3.46×10^{-16}
Z axis	38	4.71×10^{-6}	3.11×10^{-9}	-1.45×10^{-12}	2.77×10^{-16}

* Coefficient of Linear Thermal Expansion $\Delta L/L_o$

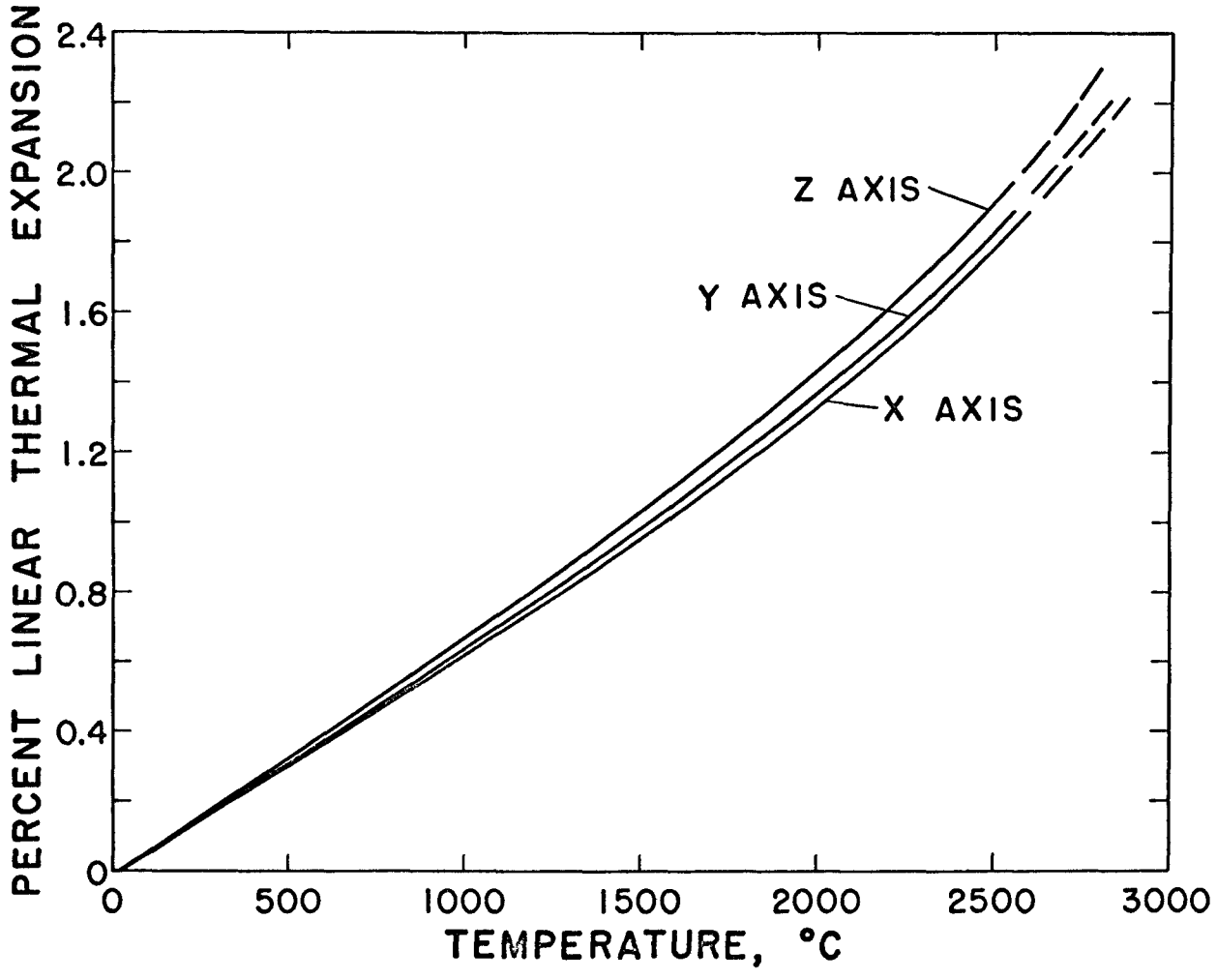


Figure 11. Percent Linear Thermal Expansion of SX-5 Graphite in Orthogonal Axes

IV. DISCUSSION

The techniques and equipment demonstrably produced thermal diffusivity values for the "round robin" Armco iron with a precision of ± 5% and an agreement of ± 3% with other published values. The thermal diffusivity of SX-5 graphite was measured over a higher temperature range by the same methods under the hypothesis that the precision and accuracy would not differ from that demonstrated on Armco iron. Multiple measurements on many samples from 800° to 2800°C in vacuum and in helium and a sophisticated least squares computer program and statistical analysis verified these statements:

1. Equal thermal diffusivity values are obtained for SX-5 graphite under a vacuum or in a helium atmosphere.
2. Different slabs of SX-5 graphite exhibit significant variations in thermal diffusivity.
3. The thermal diffusivities in the orthogonal axes from one slab of SX-5 graphite are statistically identical in the X and Y orientations; the thermal diffusivity in the Z axis is significantly different.
4. Thermal expansion coefficients in the orthogonal axes of one slab of SX-5 graphite show the same thermal anisotropy, i.e., X and Y axes equal, Z axis different.
5. The thermal diffusivity-temperature relationship can be expressed by an equation of the form $\alpha = A + B/T$ with an experimental precision of ± 5%.

The accuracy of the thermal diffusivities reported in this paper could not be compared directly with other thermal diffusivities on SX-5 graphite because none have been published. However, these thermal diffusivities were converted to thermal conductivities by eq. (4) and compared to Wagner's data [6]. In this case, our computed conductivities on SX-5 were about 25% lower than Wagner's observed values. This comparison is not too significant because the test specimens were cut from different slabs and differences in thermal properties among slabs have been demonstrated already.

A better approach to the accuracy determination compares the measured thermal conductivity and the computed conductivity on the same sample of AAQ graphite. P. Wagner kindly supplied a sample of his tested material [12] for our thermal diffusivity determinations. His thermal expansion data, Spence's [11] equation,

$$C_p = 0.4439 + 0.3079 \times 10^{-4} T - 0.6126 \times 10^{5} T^{-2} + 0.1080 \times 10^{8} T^{-3}, \qquad (7)$$

for the specific heat, and our thermal diffusivity data yield thermal conductivity data which are plotted on Figure 12.

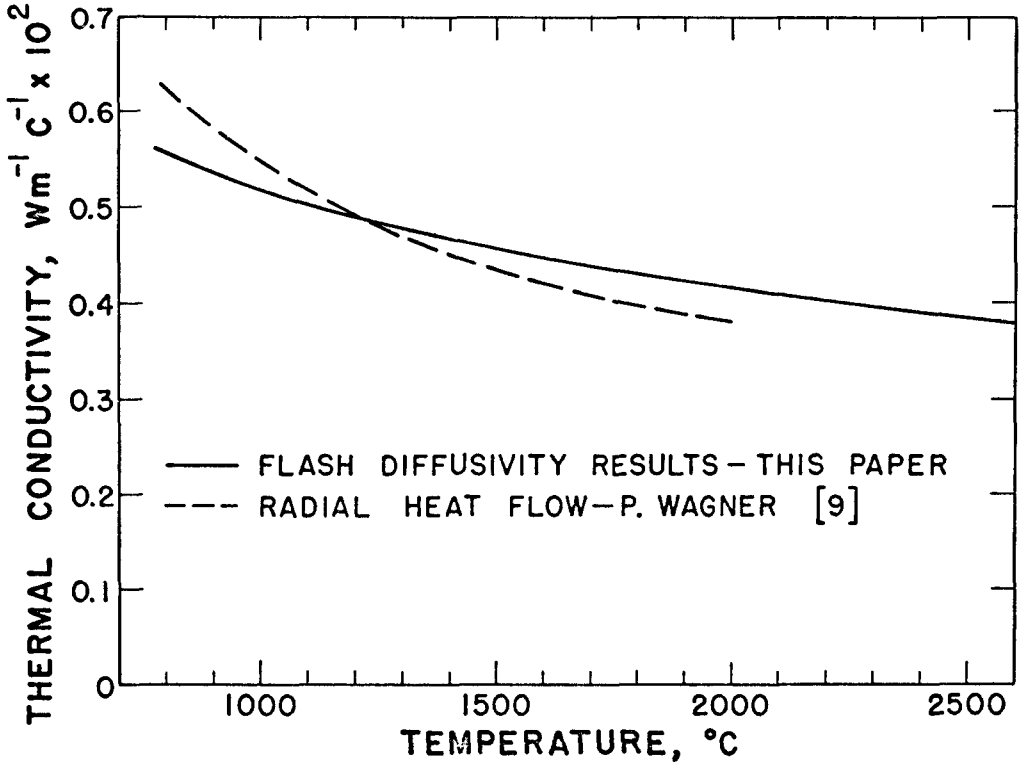


Figure 12. Thermal Conductivity of AAQ Graphite by the Radial Heat Flow and Flash Diffusivity Methods

The curves in Figure 12 indicate reasonable agreement between the thermal conductivity-temperature relationships obtained by the two methods, at least to ± 10%. Therefore, this is one verification of our hypothesis that extrapolating the accuracy of Armco iron measurements to other materials at high temperature is valid.

In conclusion, the flash method for thermal diffusivity is capable of detecting small thermal anisotropies in graphite to very high temperatures. The method is adaptable to small samples in various atmospheres, requires relatively simple equipment, and uses an uncomplicated mathematical formulation.

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