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ABSTRACT

Fundamental measurements have been obtained in the INL Graphite Characterization Laboratory to deduce the temperature dependence of thermal conductivity for G-348 isotropic graphite, which has been used by City College of New York in thermal experiments related to gas-cooled nuclear reactors. Measurements of thermal diffusivity, mass, volume and thermal expansion were converted to thermal conductivity in accordance with ASTM Standard Practice C781-08. Data are tabulated and a preliminary correlation for the thermal conductivity is presented as a function of temperature from laboratory temperature to 1000C.

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CONTENTS

FIGURES

TABLES

1. INTRODUCTION

The ability to conduct heat through a graphite core is critical to the passive removal of decay heat in typical gas-cooled nuclear reactors (GCRs) which use graphite as a moderator. Reduction of the thermal conductivity within graphite can significantly affect the passive heat removal rate and thus the peak temperature that the core and, subsequently, the fuel particles will experience during off-normal events. Determining changes to the conductivity as a function of irradiation dose and temperature is important for high-temperature gas-cooled reactor (HTGR) safety analyses.

For fundamental heat transfer experiments to examine the coupled thermal phenomena occurring in a GCR core, it is convenient to use an isotropic form of graphite. One example that has been used recently for such experiments [Valentin et al., NuReTH, 2015] is G-348 graphite from Tokai Carbon USA. This grade is described as being fine/ultrafine grain isostatic graphite. It is fabricated by cold hydrostatic pressure molding (aka "rubber press"); the result is fine grain, high density graphite with uniform structure.

In order to supplement the vendor's technical information to cover the range of coupled heat transfer data by Valentin et al., thermal properties of graphite Type G-348 were measured to deduce the temperature dependence of thermal conductivity. These measurements were obtained to assist data reduction from the experiments and to provide fundamental property data needed to employ the measurements for the assessment of computational thermal fluid mechanics codes treating GCRs. Facilities of the INL Graphite Characterization Laboratory were employed [Swank et al., INL/EXT-09-15515] and ASTM Standard Practice C781-08 for testing graphite and boronated graphite materials for high-temperature gas-cooled nuclear reactor components [2014] was followed. Direct measurements included dimensions and weight at room temperature plus thermal diffusivity and thermal expansion from room temperature to 1000 C. Thermal conductivity was then calculated from the definition of the thermal diffusivity,

$$\alpha = k / (\rho\, c_p)$$

and results have been correlated approximately for application.

2. THERMAL DIFFUSIVITY MEASUREMENTS

The measurements were performed on small thin disk-shaped specimens in accordance with ASTM Standard E1461-01. A Netzsch LFA 457 Laser Flash Apparatus was employed. A pulsed laser was used to subject one surface of a specimen to a high-intensity, short duration energy pulse. The energy of this pulse was absorbed on the front surface of the specimen and the resulting rear face temperature rise was recorded. The thermal diffusivity was calculated from the specimen thickness and the time required for the rear face temperature to reach fifty per cent of its maximum value. The LFA is complete with vendor-developed software for instrument control and data acquisition.

Two samples of about twelve mm diameter and six mm thickness were measured over ranges from room temperature to 1000 C. At each temperature three measurements were conducted and then were averaged. The resulting mean data are presented in Figure 1 and are later listed in Table 2 with other properties. One sees there is close agreement between the two samples.

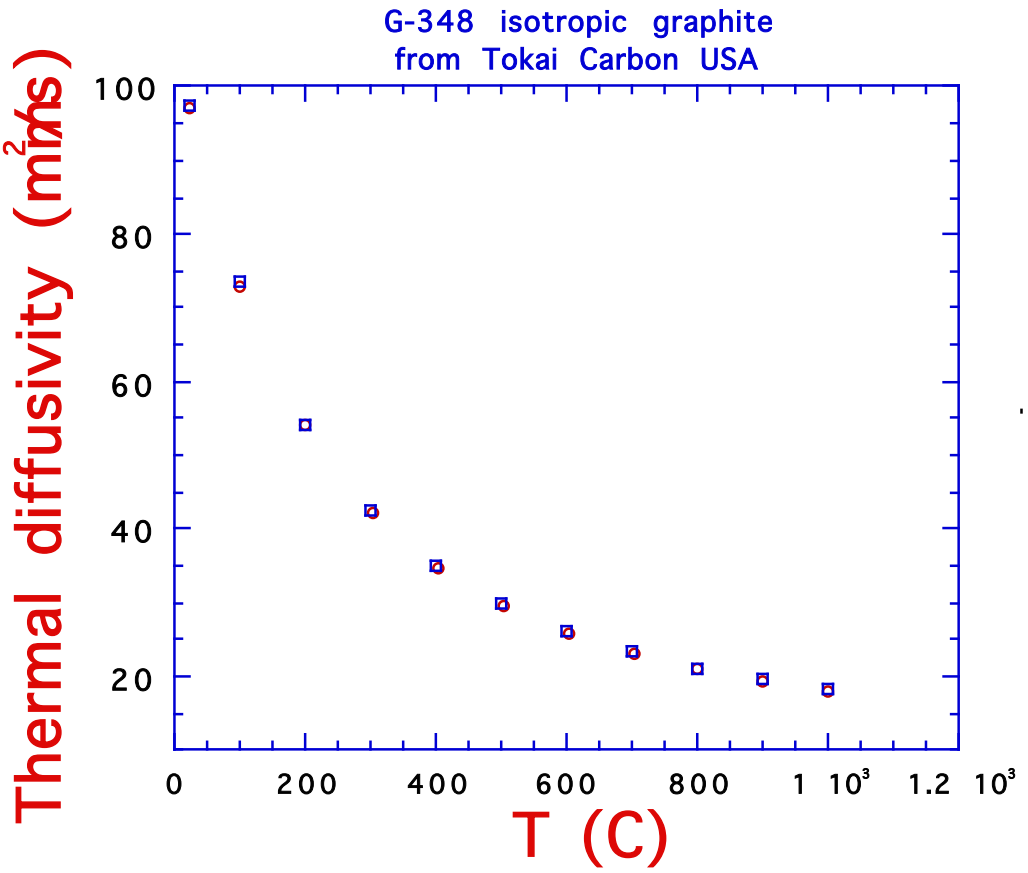

Figure 1. Measurements of thermal diffusivity. Circles = sample 1, squares = sample 2.

3. THERMAL EXPANSION MEASUREMENTS

Thermal expansion was measured in order to calculate the temperature dependence of the graphite density which appears in the definition of thermal diffusivity. In addition, dimensional change is one of the key issues affecting the performance of graphite in a neutron environment. For reactor designs and safety analyses, determination of volumetric and linear dimensional expansion as functions of temperature and radiological dose will be necessary to understand critical performance measures, such as dimensional change turnaround, irradiation creep and internal stresses imposed upon graphite components [Swank et al., 2009]. In the present study only the thermal expansion was determined.

Thermal expansion is usually quantified as a coefficient of expansion. The *coefficient of linear expansion* [Marks, pg. 293, 1916] is defined as

$$\alpha = (dL\{T\}/dT) / L\{T\}$$

and it is also called the "*instantaneous coefficient of expansion*" [Melese and Katz, eqn. 5.26, 1984]. It is a pointwise property that usually is a function of the temperature T. Often tabulated is the *mean coefficient of expansion* (eqn. 5.27 by Melese and Katz [1984]),

$$\alpha_{mean} = (L\{T\} - L\{T_{ref}\}) / ((T - T_{ref}) L\{T_{ref}\})$$

which provides an integral change in length from some reference temperature T_{ref}, typically near room or fabrication temperature.

A push rod dilatometer, Netzsch Model DIL 402 C, was applied in accordance with ASTM Standard E228-06 to determine the change in length of a graphite specimen relative to that of the holder as a function of temperature. The temperature is varied over the desired range at a slow constant heating or cooling rate starting at T_0. For the present measurements, $T_0 \approx 26$ C.

Direct measurements are the changes in length ΔL from the initial length L_0 at room temperature as the temperature T was varied. Two cylindrical samples were used. Their lengths were 24.9 mm and diameters were six mm, approximately. Table 1 provides an excerpt of recorded data over the range from 30 to 1000 C (complete measurements are available from the authors). These data were converted to the thermal expansion from a reference temperature T_{ref} and averaged as $[(L\{T\} - L_{ref}) / L_0]_{avg}$ where L_{ref} is the length at T_{ref}. These values are plotted separately for the two samples in Figure 2. In this case $T_{ref} = 30$ C.

Table 1. Thermal expansion of isotropic graphite G-348, $T_{ref} = 30$ C.

T	**$\Delta L/L_0$**	**$\Delta L/L_0$**	**$(L\{T\}-L_{ref})/L_0$**	**$(L\{T\}-L_{ref})/L_0$**	**$[(L\{T\}-L_{ref})/L_0]_{avg}$**
(C)	**Sample 3**	**Sample 4**	**Sample 3**	**Sample 4**	**Average**
30	2.39E-05	2.11E-05	0	0	0
35	4.79E-05	4.61E-05	0.000024	0.000025	0.0000245
40	7.09E-05	7.00E-05	0.000047	0.0000489	0.00004795
50	1.18E-04	1.17E-04	0.0000941	0.0000959	0.000095
60	1.64E-04	1.64E-04	0.0001401	0.0001429	0.0001415
80	2.60E-04	2.57E-04	0.0002361	0.0002359	0.000236
100	3.61E-04	3.51E-04	0.0003371	0.0003299	0.0003335
120	4.61E-04	4.47E-04	0.0004371	0.0004259	0.0004315
140	5.62E-04	5.45E-04	0.0005381	0.0005239	0.000531
160	6.65E-04	6.44E-04	0.0006411	0.0006229	0.000632
180	7.70E-04	7.47E-04	0.0007461	0.0007259	0.000736
200	8.78E-04	8.51E-04	0.0008541	0.0008299	0.000842
230	1.04E-03	1.01E-03	0.0010161	0.0009889	0.0010025
260	1.20E-03	1.18E-03	0.0011761	0.0011589	0.0011675
300	1.43E-03	1.40E-03	0.0014061	0.0013789	0.0013925
340	1.66E-03	1.62E-03	0.0016361	0.0015989	0.0016175
370	1.84E-03	1.79E-03	0.0018161	0.0017689	0.0017925
400	2.01E-03	1.96E-03	0.0019861	0.0019389	0.0019625
450	2.31E-03	2.25E-03	0.0022861	0.0022289	0.0022575
500	2.62E-03	2.54E-03	0.0025961	0.0025189	0.0025575
550	2.94E-03	2.84E-03	0.0029161	0.0028189	0.0028675
600	3.26E-03	3.14E-03	0.0032361	0.0031189	0.0031775
650	3.59E-03	3.45E-03	0.0035661	0.0034289	0.0034975
651	3.59E-03	3.45E-03	0.0035661	0.0034289	0.0034975
700	3.91E-03	3.75E-03	0.0038861	0.0037289	0.0038075

Table 1. (continued).

T	$\Delta L/L_0$	$\Delta L/L_0$	$(L\{T\}-L_{ref})/L_0$	$(L\{T\}-L_{ref})/L_0$	$[(L\{T\}-L_{ref})/L_0]_{avg}$
(C)	Sample 3	Sample 4	Sample 3	Sample 4	Average
750	4.24E-03	4.07E-03	0.0042161	0.0040489	0.0041325
800	4.57E-03	4.38E-03	0.0045461	0.0043589	0.0044525
850	4.91E-03	4.70E-03	0.0048861	0.0046789	0.0047825
900	5.25E-03	5.02E-03	0.0052261	0.0049989	0.0051125
950	5.60E-03	5.34E-03	0.0055761	0.0053189	0.0054475
1000	5.96E-03	5.66E-03	0.0059361	0.0056389	0.0057875

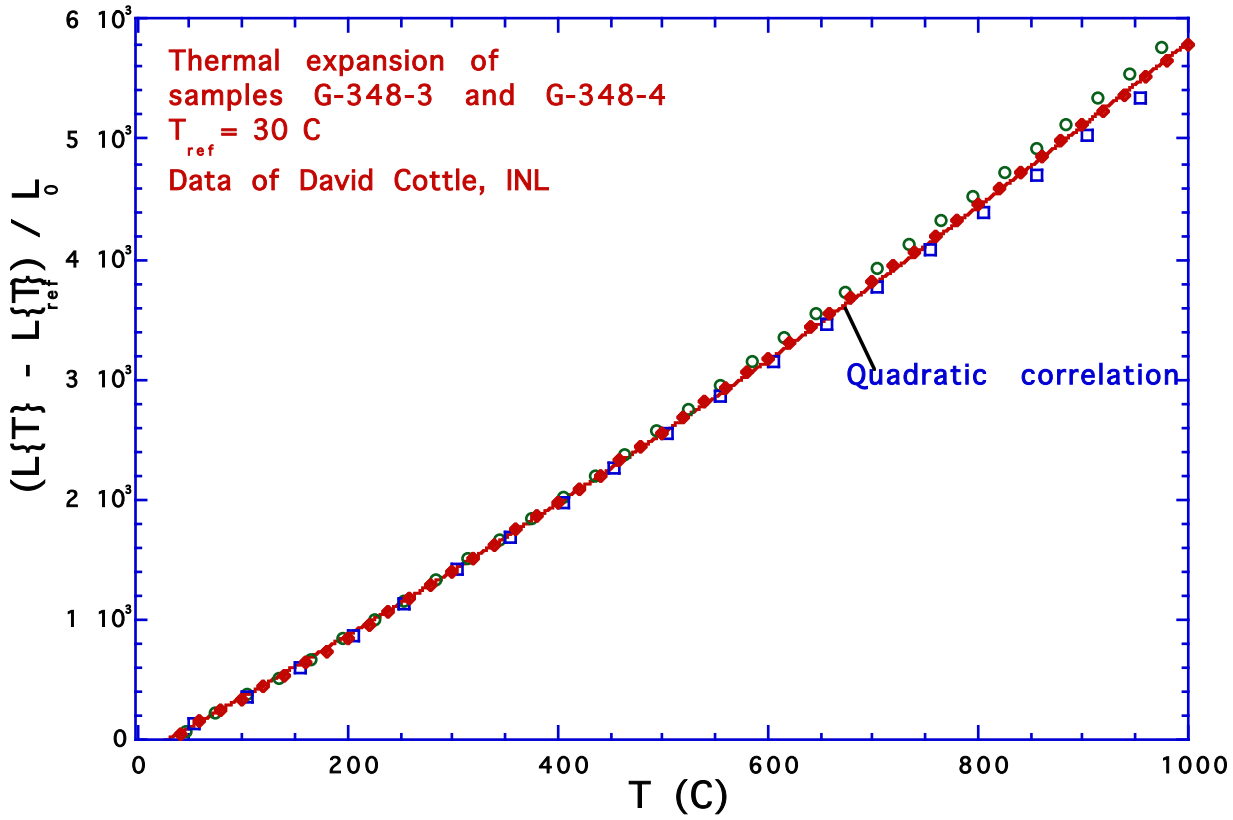

Figure 2. Thermal expansion measurements. Open symbols represent individual samples and closed diamonds are averaged values which are compared to the correlation.

The expansion can be correlated as a function of temperature and then differentiated to deduce α. We employed an approximate quadratic fit as

$$[(L\{T\} - L_{ref}) / L_0]_{avg} = a + b\,T + c\,T^2$$

which gave a = - 0.0001454, b = $4.812 \times 10^{(-6)}$ and c = $1.145 \times 10^{(-9)}$ with T in degrees C. This resulting correlation is also plotted in Figure 2. Agreement is within about five per cent at low temperatures but better than one per cent at the high temperature end of the range. It is recognized that closer agreement could probably be reached with a more sophisticated fitting procedure but, since the entire expansion at 1000 C is less than 0.6 per cent, the effect on the calculated density is small. For application in deriving expansion coefficients, this correlation may be rearranged as $(L_{ref}/L_0) = 1 - (a + bT_0 + cT_0^2)$ or $(L/L_0) = a + b\,T + c\,T^2 + (L_{ref}/L_0)$. For our measurements, (L_{ref}/L_0) is within 0.002 per cent of unity (aka "negligible"). From these relations one can calculate the change in length between two temperatures as

$$\Delta L_{1\text{-}2} = L_0 \left[b\,(T_2 - T_1) + c\,(T_2^2 - T_1^2)\right]$$

Temperature T_1 could be the initial temperature T_0, reference temperature T_{ref}, the measurement temperature after fabrication or some other appropriate temperature.

Differentiation of the correlation with respect to T gives

$$(dL\{T\}/dT) / L_0 = b + 2\,c\,T$$

This relation can be converted to the *coefficient of linear expansion*

$$\alpha = (L_0/L\{T\})\,(dL\{T\}/dT)/L_0 = (b + 2c\,T) / [1 + b\,(T - T_0) + c\,(T^2 - T_0^2)]$$

via some algebra. For the present range of measurements, the denominator is less than 0.6 per cent of unity at the maximum temperature, 1000 C, so it could be neglected in many cases. Likewise one can derive a *mean coefficient of expansion* from T_1 to T_2 to be

$$\alpha_{mean,1\text{-}2} = [b + c\,(T_2 + T_1)] / [1 + b\,(T_1 - T_0) + c\,(T_1^2 - T_0^2)]$$

Temperature T_1 can be replaced by T_{ref} or T_0 if desired. For example, a mean coefficient of expansion based on a reference temperature can be written as

$$\alpha_{mean,ref} = [b + c\,(T + T_{ref})] / [1 + b\,(T_{ref} - T_0) + c\,(T_{ref}^2 - T_0^2)]$$

For the present measurements, again the denominator is about 0.002 per cent of unity so this mean coefficient could be approximated as

$$\alpha_{mean,ref} \approx 4.812 \times 10^{(-6)} + 1.145 \times 10^{(-9)}\,(T + 30)]$$

(with $\alpha_{mean,ref}$ having units of 1/C and temperature in degrees C) for our G-348 graphite.

4. DENSITY

As noted, the graphite density is required to calculate the thermal conductivity from the thermal diffusivity measurements. It is a two-step process. First a sample's density is determined at room temperature from mass and dimension measurements. Then the density variation with temperature is calculated from the thermal expansion which has been measured and correlated as above.

Dimensional and mass measurements are performed to ASTM C559-05. This standard describes in detail the procedure for making dimensional measurements and calculating the bulk density. Dimensional measurements of specimen diameter and length are made with Mitutoyo micrometers and calipers. The mass is measured using an electronic balance, a Sartorius Scale ME235P. The micrometers, calipers and balance are all calibrated by the INL Standards and Calibration Laboratory. Measured values are transferred directly from the measurement tools into Labview software. Once the physical and dimensional measurements of the specimens are obtained the data are automatically written to an MS Excel spread sheet. These data are used to calculate initial bulk density and are available for other measurement calculations.

Density is defined as $\rho\{T\} = M/V\{T\}$ where M is the mass of the object and V is its volume. It was determined for each of the two samples employed in the thermal diffusivity measurements. Mass and volume were measured at $T_0 = 20$ C. The thermal strains at the other temperatures were deduced from the mean coefficient of expansion and the increase in temperature as

$$\varepsilon = [(L - L_0) / L_0] = \alpha_{mean,0} (T - T_0)$$

which gave the variation in volume as

$$V\{T\} = V_0 (1 + \varepsilon)^3$$

Figure 3 provides the results; the difference between the samples is about three per cent. Tabulated values are listed later in this report.

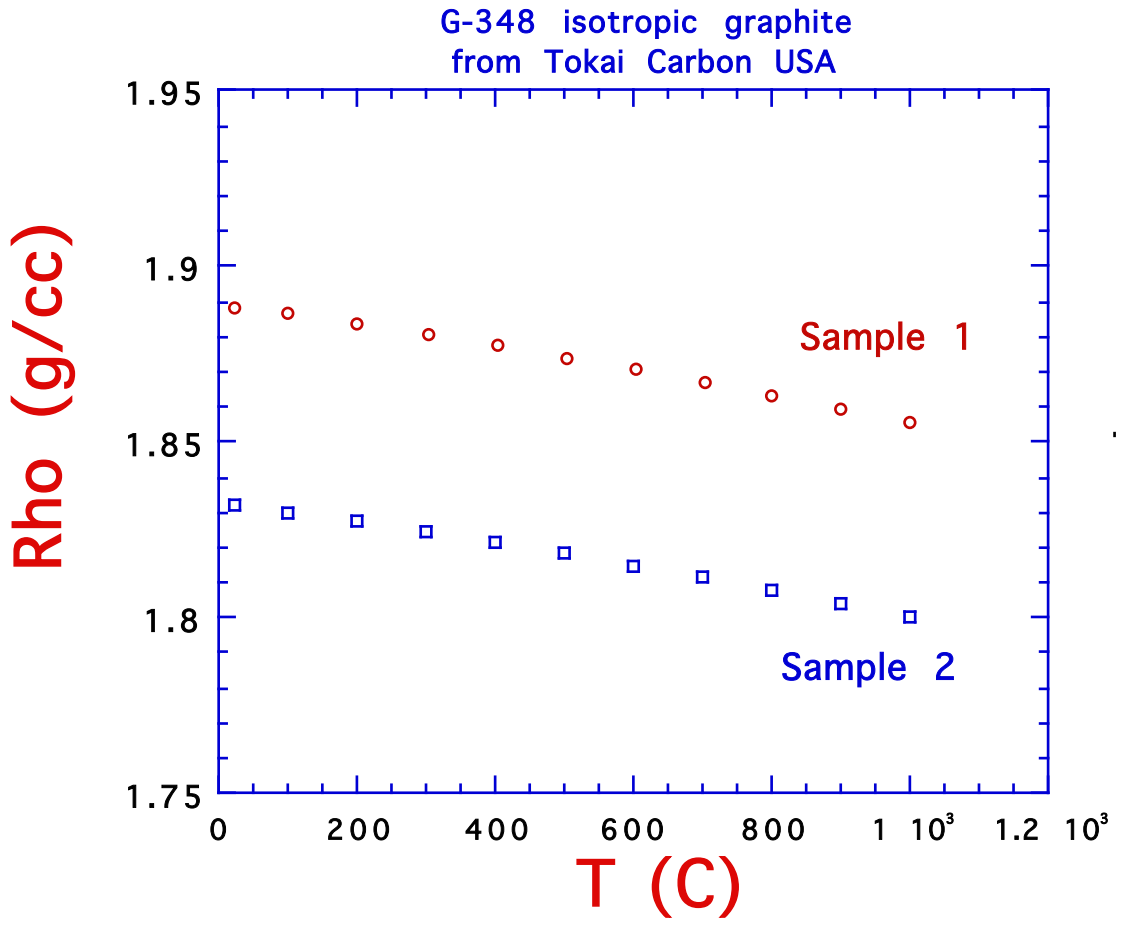

Figure 3. Temperature dependence of bulk density for G-348 graphite.

5. SPECIFIC HEAT

ASTM Standard Procedure C781-08 provides recommended values of specific heat of graphite in its Table A6.1 [2014]. A correlation for the range $300 < T < 3000$ K is also given; according to the Procedure, the equation represents the tabulated values within two per cent. The tabulated values (circles) and correlation (solid curve) are plotted in Figure 4.

Butland and Maddison [1973/4] reviewed the measurements of specific heat at constant pressure for graphite and recommended a polynomial for use with nuclear graphite. They considered their polynomial to be valid for the range 250 < T < 3000 K. The "unadjusted polynomial" of Butland and Maddison is also included for comparison in Figure 4 as a dashed curve.

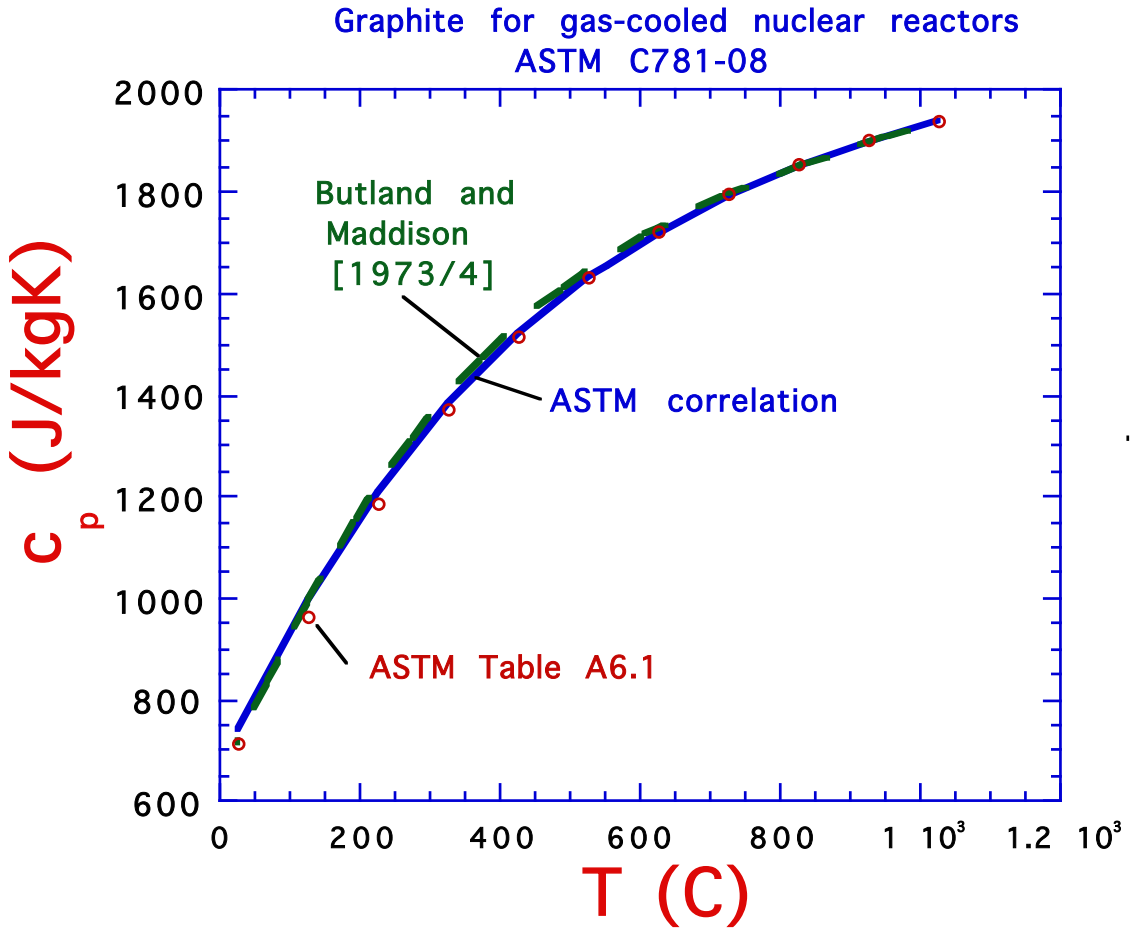

Figure 4. Recommended values of the specific heat of graphite [ASTM Standard Procedure C781-08, 2014].

6. DEDUCED THERMAL CONDUCTIVITY

The thermal conductivity k was calculated from each data point on thermal diffusivity via the definition and from the measurements and deduced values above as $k = \alpha \rho c_p$. The deduced values for each sample are listed in Table 2 along with other deduced thermal properties. Figure 5 plots the values deduced for each sample.

Table 2. Thermal properties of isotropic graphite G-348.

T C	α mm^2/s	c_p J/(kgK)	ρ g/cc	k W/(mK)	k Btu/(hrftR)
Sample G-348-1					
22.6	96.992	726.19	1.8885	133.02	76.91
101.0	73.025	933.15	1.8863	128.54	74.32
199.3	54.095	1154.47	1.8835	117.62	68.01
301.6	42.047	1341.07	1.8804	106.03	61.30
401.6	34.646	1486.83	1.8772	96.70	55.91
501.6	29.489	1603.53	1.8739	88.61	51.23
601.6	25.896	1697.43	1.8705	82.22	47.54
701.7	23.109	1773.60	1.8670	76.52	44.24
801.3	20.985	1835.58	1.8634	71.78	41.50
900.8	19.347	1886.68	1.8596	67.88	39.25
1000.9	17.947	1929.44	1.8557	64.26	37.15
Sample G-348-2					
23.5	97.368	728.71	1.8322	130.00	75.16
101.1	73.429	933.40	1.8301	125.43	72.52
200.6	54.235	1157.11	1.8273	114.67	66.30
300.8	42.562	1339.77	1.8243	104.03	60.15
401.0	34.944	1486.04	1.8213	94.57	54.68
500.9	29.873	1602.80	1.8181	87.05	50.33
601.0	26.098	1696.92	1.8148	80.37	46.47
701.1	23.356	1773.19	1.8114	75.02	43.37
800.9	21.073	1835.36	1.8078	69.92	40.43
900.7	19.590	1886.63	1.8042	66.68	38.55
1000.7	18.167	1929.36	1.8004	63.11	36.49

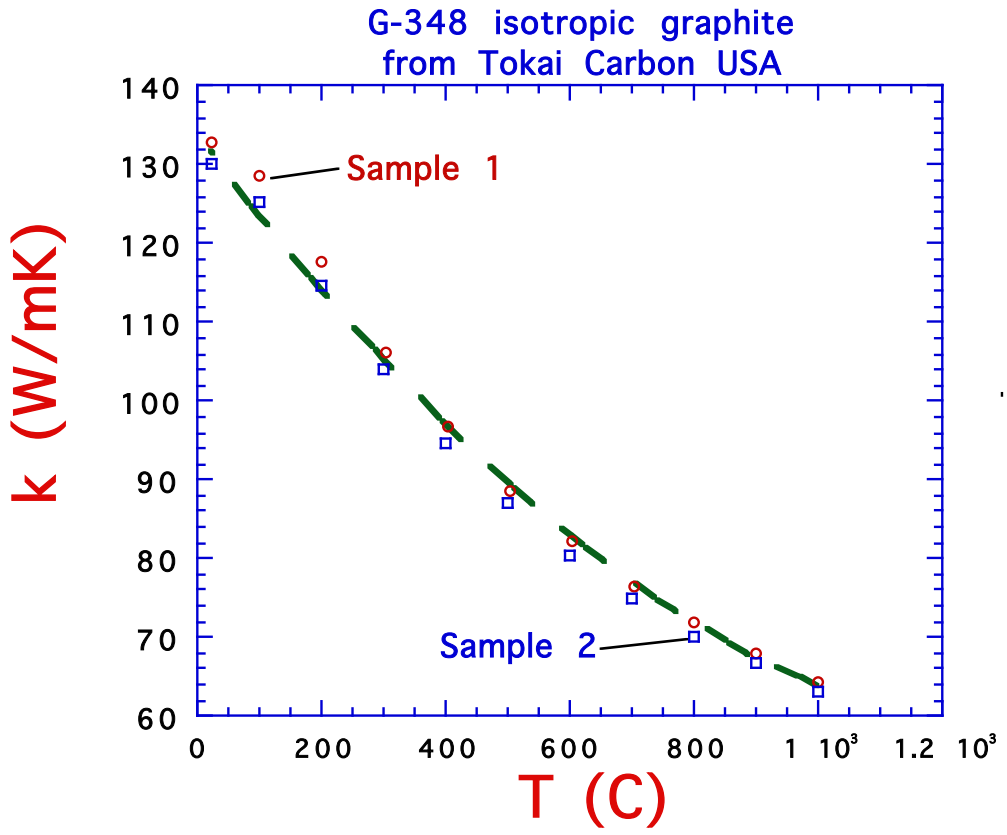

Figure 5. Temperature dependence of thermal conductivity for G-348 graphite. Sample 1 = circles, sample 2 = squares and quadratic correlation = dashed curve.

For application in computer calculations, the averaged values of the thermal conductivity of the G-348 samples have been approximated by a simple quadratic relation,

$$k\ (W/mK) = 134.0 - 0.1074\ T + 3.719 \times 10^{(-5)}\ T^2$$

with T in degrees C. It is included in Figure 5 as a dashed curve. This correlation agrees with the averaged values to within two per cent except in the vicinity of 100 C. It is recognized that with more sophisticated relations the agreement probably could be improved.

7. ESTIMATED EXPERIMENTAL UNCERTAINTIES

Estimates of the experimental uncertainties of the instrumentation and their measurements have been collected from a variety of sources, such as vendors, the internet, the INL Standards and Calibration Laboratory, etc. This collection of estimates is presented as Table 3 which includes citations of the sources of the individual estimates. Following the guidance of Kim, Simon and Viskanta [1993], we have separated these estimates into systematic uncertainties (also called "bias" uncertainties) and random uncertainties (or "precision" uncertainties). For understanding uncertainty estimation for measurements and deduced results, the reader is referred to the publications of Kline and McClintock [1955], Moffat [1982, 1988], Kline [1985], Lassahn [1985] and Abernethy, Benedict and Dowdell [1989] and to the text of Taylor [1997].

Table 3. Estimated experimental uncertainties of measurements.

	Systematic (bias) Uncertainty	Random Uncertainty	Source
Thermal Diffusivity			
Netzsch LFA 457			www.netzsch-thermal
Laser flash apparatus			-analysis.com/us/
"Repeatability"		2%	products-solutions/
"Accuracy"	3%		thermal-diffusivity - conductivity/lfa-457-microflash/ "Data sheet"
Lengths			
Mitutoyo Digimatic Bench			W. Chan, Mitutoyo
Micrometer 121-155			e-mail 22 Mar 2016
"Resolution"		0.001 mm	
"Accuracy"	0.0001 in.		
"Resolution"		0.001 mm	User's Manual No.
"Instrument error"	2 µm		1033, Series 121,
"Origin point error"	3 µm		Mitutoyo
Calibration			INL Standards and Calibration Lab.
"Tolerance"	0.00005 in.		
Machining thermal diffusivity samples	0.5%		W. D. Swank, INL, e mail 5 Aug 2015
Mass			
Sartorius ME 235 P			scaleman.com/
analytical balance			analytical-digital-
"Readability"		0.01 mg	electronic-balance-
"Repeatability"		0.015 mg	me235p.html
"Linearity"	0.15 mg		
"Sensitivity drift"		10 ppm/C	
Calibration			INL Standards and
"Repeatability"			Calibration Lab.
Standard deviation"		19 µg	
"Readability"		10 µg	
"Temperature drift sensitivity"		60 µg	
"Linearity (at 1 g)"	28 µg		
"Weight uncertainty"	45 µg		
Thermal Expansion			
Netzsch dilatometer			
DIL 402 C			M. Tucker, Netzsch,

Table 3. (continued).

	Systematic (bias) Uncertainty	Random Uncertainty	Source
"Uncertainty"	3%		e-mail 5 April 2016
"Temperature accuracy"	<0.5 K		
"Temperature precision"		0.01 K	
"Temperature resolution (digital)"		0.001 K	
"Dl resolution"		0.125 nm 1.25 nm/digit	M. Tucker, Netzsch, e-mail 7 April 2016
"Noise of Δl			
peak-to-peak		<10 nm	
RMS"		<2 nm	
"Dl drift			
at 200 C		<1 μm/h	
at 1200 C"		<2.5 μm/h	
c_p Correlation	2.0%		ASTM SP C781-08 Section A6

In general, the sources have not provided quantitative confidence estimates such as "odds" nor have they indicated the number of standard deviations from a mean that they represent [Taylor, p. 135, 1997]. In the fields of heat transfer and fluid mechanics, estimated uncertainties are usually based on estimates of confidence limits of about 95 per cent (20:1 odds) [Kim, Simon and Viskanta, 1993]. For the purposes of this report, we assume that claims of "uncertainty" or "accuracy" represent 20:1 odds or approximately two standard deviations; other claims, such as "instrument error," "sensitivity" and such, are assumed to correspond to one standard deviation.

Following the guidance of Kline and McClintock [1955], Taylor [1997] and others for propagation of uncertainties, we estimated the combined uncertainties of the individual measurements. For calculating the estimated uncertainty in deduced thermal conductivity, we take the systematic uncertainty in c_p as two per cent in accordance with ASTM SP C781-08 and neglect its random uncertainty. For the dimensions measured at room temperature, the uncertainty in machining the samples dominates the systematic uncertainty. The linearity specification dominates the systematic uncertainty for mass measurements and the calibration showed the temperature drift sensitivity to dominate their random uncertainty. For the thermal expansion, the dominant random uncertainty is due to the drift which could have ranged from about two to twenty μm over the six hour course of the measurement; the larger values correspond to the higher temperatures. The resulting estimates of the individual measurement uncertainties are summarized as follows:

	Systematic Uncertainty	Random Uncertainty
Thermal diffusivity	3%	2%
Lengths	0.5%	0.001 mm
Mass	150 μg	120 μg
Thermal expansion	3%	2 – 20 μm
Temperature	0.5 K	0.02 K

For the properties involved in the calculations of the thermal conductivities, these individual measurement uncertainties propagate into the following estimates of the uncertainties in the properties:

	Systematic Uncertainty	Random Uncertainty
Temperature, T	0.5 K	0.02 K
Thermal diffusivity, α	3%	2%
Density, ρ (100 C)	1.1%	0.07%
Density, ρ (1000 C)	1.1%	0.46%
Specific heat, c_p	2%	—

Thus, the systematic uncertainty for the thermal conductivity ($k = \rho\, c_p\, \alpha$) of each sample becomes about 3.8 per cent and its random uncertainty is estimated to be about two per cent. For the averages of the two samples, the systematic uncertainty then is about 2.7 per cent and the random uncertainty is approximately 1.5 per cent [Taylor, sec. 4.4, 1997].

8. CONCLUDING REMARKS

Fundamental measurements have been obtained in the INL Graphite Characterization Laboratory to deduce the temperature dependence of thermal conductivity for G-348 isotropic graphite, which has been used by City College of New York in thermal experiments related to gas-cooled nuclear reactors. Mass and volume were measured at laboratory temperature and thermal diffusivity data were obtained from laboratory temperature to 1000 C for two samples.

Two additional samples were employed to measure thermal expansion, also from laboratory temperature to 1000 C. Averaged values of the thermal strain were correlated as

$$[(L\{T\} - L_{ref}) / L_0]_{avg} = - 0.0001454 + 4.812 \times 10^{(-6)}\, T + 1.145 \times 10^{(-9)}\, T^2$$

with T in degrees C. Agreement with the measurements is within about five per cent at low temperatures but better than one per cent at the high temperature end of the range. From this relation, expressions for the coefficient of linear expansion and mean coefficients of expansion were deduced. The temperature dependence of the density was then calculated from these thermal expansion data.

To calculate thermal conductivity from the definition, $k = \rho\, c_p\, \alpha$, ASTM Standard Practice C781-08 provides recommended values of specific heat and a correlation versus temperature. From $c_p\{T\}$, the deduced $\rho\{T\}$ and thermal diffusivity data, thermal conductivity was calculated for each of the two examples. Averaged values were correlated by the relation

$$k\ (W/mK) = 134.0 - 0.1074\, T + 3.719 \times 10^{(-5)}\, T^2$$

with T in degrees C. Agreement with the averaged values is within two per cent except in the vicinity of 100 C. It is recognized that agreement probably could be improved with a more sophisticated correlation relation, perhaps as suggested by Churchill and Usagi [AIChE J. 1972], but this correlation is a significant improvement over the information available on the vendor's web site. Estimated experimental uncertainties in the averaged thermal conductivity are approximately 2.7 (or three) per cent for the systematic uncertainties and about 1.5 per cent for random uncertainties.

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