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Heat Capacity and Electrical Resistivity of POCO AXM-5Q1 Graphite in the Range 1500–3000 K by a Pulse-Heating Technique

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Measurements of the heat capacity and electrical resistivity of POCO AXM-5Q1 graphite in the temperature range 1500–3000 K by a subsecond-duration pulse-heating technique are described. The results for heat capacity may be represented by the relation

$$C_p = 19.438 + 3.6215 \times 10^{-3}\, T - 4.4426 \times 10^{-7}\, T^2$$

where C_p is in $J \cdot mol^{-1} \cdot K^{-1}$ and T is in K. The results for electrical resistivity vary with the density (d) of the specimen material and, therefore, are represented by the following relations: for $d = 1.709$,

$$\rho = 1084.6 - 1.9940 \times 10^{-1}\, T + 1.6760 \times 10^{-4}\, T^2 - 2.4896 \times 10^{-8}\, T^3$$

and for $d = 1.744$,

$$\rho = 943.1 - 1.3836 \times 10^{-1}\, T + 1.3776 \times 10^{-4}\, T^2 - 2.0310 \times 10^{-8}\, T^3$$

where ρ is in $\mu\Omega \cdot cm$, T is in K, and d (at 20°C) is in $g \cdot cm^{-3}$. The maximum uncertainties in the measured properties are estimated to be 3% for heat capacity and 1% for electrical resistivity.

KEY WORDS: electrical resistivity; graphite; heat capacity; high temperatures; pulse-heating technique.

1. INTRODUCTION

In recent years, graphite has become one of the most important refractory materials in the development of high-temperature technologies, finding applications as a thermal protective material, as a component in structural

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composites, etc. It also has the potential of becoming a reference material for thermal property measurements. As a result, there has been an increasing need for accurate data on various thermophysical properties of graphite, particularly at temperatures above 2000 K, where significant disagreements exist in the literature.

In the present study, accurate measurements of the heat capacity and electrical resistivity of POCO AXM-5Q1 graphite were conducted in the temperature range 1500 to 3000 K, as part of the CODATA program on properties of high-temperature reference materials. The experiments were performed by means of a rapid pulse-heating technique which involves resistively heating the specimen from room temperature to temperatures above 1500 K by passing a subsecond electrical current pulse through it and simultaneously measuring (with millisecond resolution) pertinent experimental quantities such as the specimen temperature, voltage drop across the specimen, and current through the specimen. The short heating time (less than 1 s) of this technique tends to minimize problems associated with quasi-steady-state experiments at high temperatures, in particular those created by the increased heat transfer and by specimen evaporation.

Details regarding the construction and operation of the measurement system, the methods of measuring experimental quantities, and other pertinent information such as the formulation of relations for determining properties, error analyses, etc., are given in earlier publications [1, 2].

2. MEASUREMENTS

2.1. Specimens

The specimen material was provided by the Office of Standard Reference Materials (OSRM) at the NBS from the lot of POCO AXM-5Q1 graphite, which is being considered as a candidate reference material for thermal conductivity and other thermophysical measurements at high temperatures. The graphite was supplied in the form of two cylindrical rods which were identified by the OSRM as rod 0045 (30 cm long) and as a portion (10 cm long) from rod 0046 near "end 1." The bulk densities (at 293 K) of graphite rods 0045 and 0046 were reported by OSRM to be 1.744 and 1.709 $g \cdot cm^{-3}$, respectively.

The sample rods were used to fabricate three specimens labeled as follows: specimen 1, from the 10-cm portion of rod 0046; and specimens 2 and 3, from rod 0045 near "end 2." Each specimen was fabricated into the form of a tube having the following nominal dimensions: length, 76 mm; outside diameter, 6.4 mm; and wall thickness, 0.5 mm. A small rectangular sighting hole (0.5×1 mm) was fabricated through the wall at the middle of the tube, thereby approximating a blackbody cavity for the pyrometric temperature measurements. In order to compensate for the cross-sectional nonuniformity created by the hole, a portion of the specimen was removed by grinding a flat along the length of the tube excluding the 1 mm length of the hole.

2.2. Procedure

In order to optimize the operation of the high-speed photoelectric pyrometer [3], the temperature interval of the measurements (1500–3000 K) was divided into six overlapping temperature ranges. One pulse-heating experiment per specimen was performed in each temperature range. All experiments were conducted with the specimen in a vacuum environment at approximately 1.3 mPa ($\sim 10^{-5}$ torr).

Prior to each experiment, the voltage of the battery bank and the value of the resistance in series with the specimen were both adjusted in order to achieve the desired heating rate in a given temperature range. Each specimen was pulse heated successively from room temperature through each temperature range, beginning with the lowest range. The heating rates were varied, depending upon the temperature range, between 3000 and 5000 $K \cdot s^{-1}$. The duration of the electrical current pulse, used to resistively heat the specimen, ranged between 400 and 500 ms.

To study the possible effects that may be attributed to the rate at which the specimen heats, 16 additional experiments in the temperature range 1900–2100 K (Range III) were conducted on specimen 3, using heating rates between 600 and 6500 $K \cdot s^{-1}$. The results of these experiments (see Section 3.3) were not used in the final computation of the heat capacity and electrical resistivity of POCO AXM-5Q1 graphite.

Upon completion of the experiments, the high-speed pyrometer was calibrated against a tungsten-filament reference lamp which, in turn, had been calibrated against the NBS photoelectric pyrometer by the Radiometric Physics Division at the NBS. All temperatures reported in this work are based on the International Practical Temperature Scale of 1968 [4].

3. RESULTS

The data on temperature, current, and voltage within each temperature range were fitted by polynomial functions for each quantity in terms of time by means of the least-squares method. The functions were then used to compute the values of heat capacity and electrical resistivity corresponding to each experiment; the results are given in Tables AI and

AII in the Appendix. The final values for the properties were obtained as described below; they are presented at 100 K temperature intervals in Table I. It should be noted that, in all computations, the geometrical quantities of the specimen are based on their room-temperature (295 K) dimensions.

3.1. Heat Capacity

The heat capacity was computed from data taken during the heating period by means of the relation $C_p = (ei - P_r)/nT'$, where e is the voltage drop across the specimen, i the current, P_r the power loss due to thermal radiation, n the number of moles, and T' the heating rate of the specimen. The correction for radiative heat loss was calculated on the basis of data taken during the initial free cooling of the specimen, following the heating period during the same experiment. Typical values for this correction are given as $(P_r/ei) \times 100\%$ in Table II. Results in the six temperature ranges for the three specimens (see Table AI) were combined and fitted by a

Table I. Smoothed Heat Capacity and Electrical Resistivity of POCO AXM-5Q1 Graphite

Temperature (K)	Heat capacity ($J \cdot mol^{-1} \cdot K^{-1}$)[a]	Electrical resistivity ($\mu\Omega \cdot cm$)	
		Specimen 1[b]	Specimens 2 & 3[c]
1500	23.87	1078.6	977.0
1600	24.10	1092.6	991.2
1700	24.31	1107.7	1006.2
1800	24.52	1123.5	1021.9
1900	24.72	1140.0	1038.2
2000	24.90	1157.0	1054.9
2100	25.08	1174.4	1072.0
2200	25.26	1192.0	1089.2
2300	25.42	1209.7	1106.5
2400	25.57	1227.3	1123.8
2500	25.72	1244.6	1140.9
2600	25.85	1261.6	1157.7
2700	25.98	1278.0	1174.0
2800	26.10	1293.7	1189.9
2900	26.20	1308.7	1205.1
3000	26.30	1322.6	1219.5

[a] Heat capacity values according to Eq. (1).
[b] Electrical resistivity values according to Eq. (2).
[c] Electrical resistivity values according to Eq. (3).

Table II. Typical Values of the Radiative Heat-Loss Correction for Pulse-Heating Experiments on POCO AXM-5Q1 Graphite

Temperature (K)	Radiative heat loss (% of input power)
1500	4
2000	10
2500	19
3000	34

quadratic function in temperature by means of the least-squares method. The function that represents the final results (standard deviation = 0.4%) for the heat capacity of POCO AXM-5Q1 graphite at temperatures between 1500 and 3000 K is

$$C_p = 19.438 + 3.6215 \times 10^{-3} T - 4.4426 \times 10^{-7} T^2 \qquad (1)$$

where C_p is in $J \cdot mol^{-1} \cdot K^{-1}$ and T is in K. In the computations of heat capacity, the atomic weight of graphite was taken as 12.011. The deviation

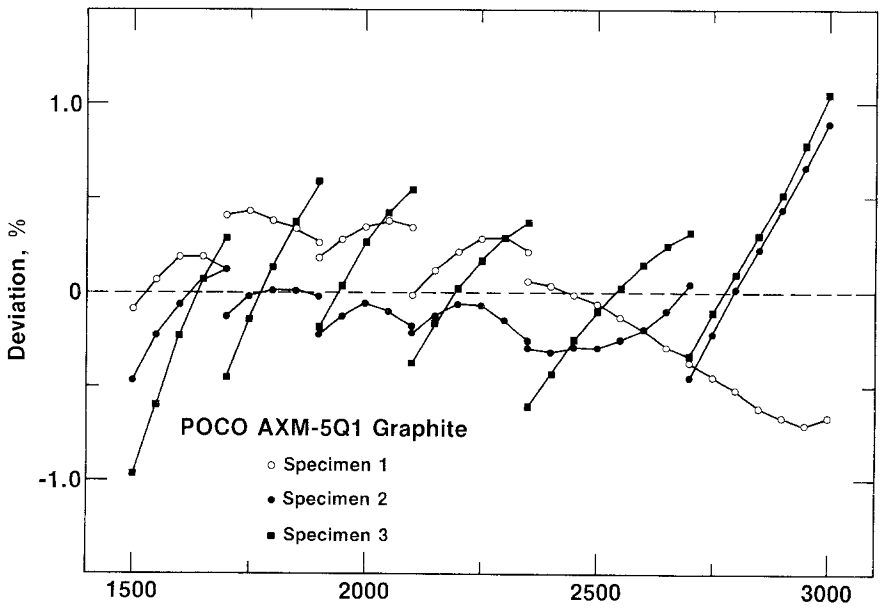

Fig. 1. Deviation of heat capacity results for three specimens of POCO AXM-5Q1 graphite from the smooth function given by Eq. (1).

of the heat-capacity values for the three specimens from the smooth function defined by Eq. (1) is shown in Fig. 1. As may be seen, the deviations are about 0.5% or less at all but the highest temperatures, where the deviations become as large as 1%.

3.2. Electrical Resistivity

The electrical resistivity of the specimen was computed by means of the relation $\rho = RA/L$, where R is the resistance, A the cross-sectional area, and L the length of the specimen between the voltage probes. The cross-sectional area was obtained from the density of the graphite material and the measurement of the specimen weight. The respective densities used in the computations were 1.709 $g \cdot cm^{-3}$ for specimen 1 (from rod 0046) and 1.744 $g \cdot cm^{-3}$ for specimens 2 and 3 (from rod 0045) as reported by the OSRM. As a check, the density of the portion of rod 0045 near "end 1" was measured, yielding the value 1.745 $g \cdot cm^{-3}$. Considerable differences (~10%) in resistivity values were obtained for specimens with different densities. Therefore, the results for specimen 1 and those for specimens 2 and 3 (see Table AII) where fitted separately by cubic polynomials in tem-

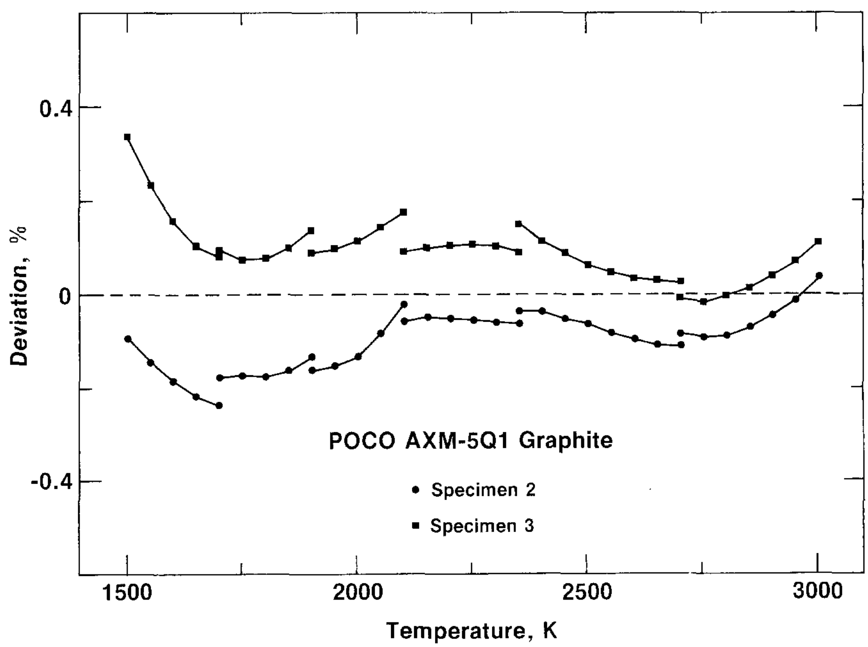

Fig. 2. Deviation of electrical resistivity results for two specimens of POCO AXM-5Q1 graphite from the smooth function given by Eq. (3).

perature by means of the least-squares method. The functions which represent the final results for the electrical resistivity of POCO AXM-5Q1 graphite in the temperature range 1500 to 3000 K are as follows.

For specimen 1 (standard deviation = 0.1 %),

$$\rho = 1084.6 - 1.9940 \times 10^{-1} T + 1.6760 \times 10^{-4} T^2 - 2.4896 \times 10^{-8} T^3 \quad (2)$$

and for specimens 2 and 3 (standard deviation = 0.2 %),

$$\rho = 943.1 - 1.3836 \times 10^{-1} T + 1.3776 \times 10^{-4} T^2 - 2.0310 \times 10^{-8} T^3 \quad (3)$$

where ρ is in $\mu\Omega \cdot$cm and T is in K. Figure 2 presents the deviation of the electrical resistivity values for specimens 2 and 3 from the smooth function defined by Eq. (3). Prior to the pulse experiments, a Kelvin bridge was used to measure the electrical resistivity of specimens 1, 2, and 3 at "room temperature" (293 K), yielding values of 1735, 1533, and 1545 $\mu\Omega \cdot$cm, respectively.

The electrical resistivity of graphite passes through a minimum as it is heated from room temperature to temperatures in the present range, as may be seen in Fig. 3. The minimum values for specimens 1 and 3 were determined to be 1065 and 960 $\mu\Omega \cdot$cm, respectively.

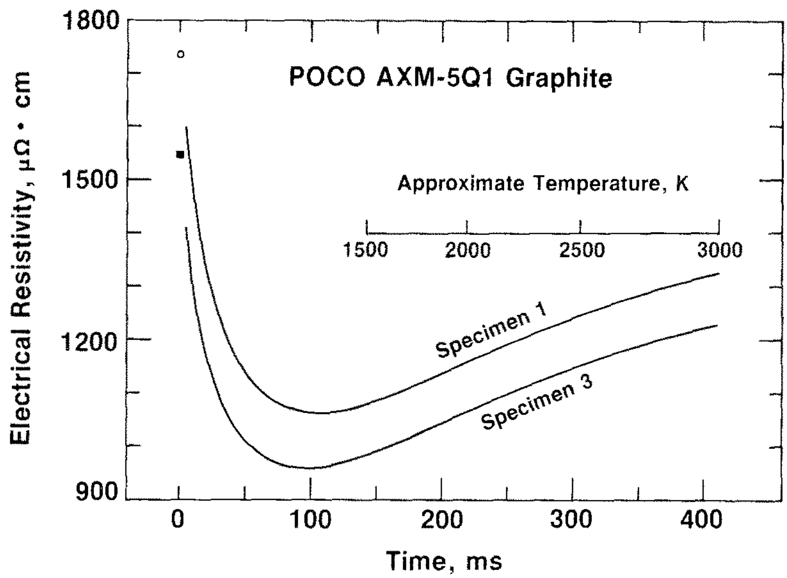

Fig. 3. Electrical resistivity of POCO AXM-5Q1 graphite as a function of time for experiments in which the specimen is pulse heated from "room temperature" (293 K) to 3000 K. An approximate temperature scale (nonlinear) is shown for the latter part of the heating.

3.3. Estimate of Errors

The details for estimating the errors in measured and computed quantities in high-speed experiments with the present measurement system are given in an earlier publication [2]. Specific items in the error analyses were recomputed whenever the present conditions differed from those in the earlier publication. The results on the imprecision[2] and uncertainty[3] of the measured and computed quantities are given in Table III.

In order to determine the effect of the specimen heating rate on the measurement of heat capacity and electrical resistivity, 16 pulse experiments with heating rates ranging from about 500 to 6500 $K \cdot s^{-1}$ were performed on specimen 3; the results at 2000 K are presented in Fig. 4. As may be seen, the results for heat capacity appear to be relatively insensitive to heating rates in this range, whereas the values for electrical resistivity begin to decrease as the heating rate is decreased below 2000 $K \cdot s^{-1}$. The maximum difference in heat capacity, corresponding to the range 500–6500 $K \cdot s^{-1}$, and in electrical resistivity, corresponding to the range 2000–6500 $K \cdot s^{-1}$, is in both cases approximately 0.2%, which is about the same as the imprecision of the measurements. Since the experiments (other than the above 16) conducted in the present work correspond to heating rates of approximately 3500 $K \cdot s^{-1}$ (at 2000 K), the reported final results are not dependent on the specimen heating rate.

When a specimen heats at a low rate, temperature gradients are established in the axial direction due to heat conduction from the specimen to the clamps. As a result, the average temperature of the specimen will be lower than the measured temperature (based on thermal radiation from the

[2] Imprecision refers to the standard deviation of an individual point as computed from the difference between the measured value and that from the smooth function obtained by the least-squares method.

[3] Uncertainty refers to the estimated maximum total error (random and systematic). Sometimes it is also referred to as "inaccuracy."

Table III. Summary of the Error Analysis[a]

Quantity	Imprecision	Uncertainty
Temperature (at 2000 K) (K)	0.4	5
Current (%)	0.03	0.1
Voltage (%)	0.02	0.1
Radiative heat-loss correction (%)	1	3
Heat capacity (%)	0.5	3
Electrical resistivity (%)	0.2	1

[a] Definitions of the terms imprecision and uncertainty are given in the text.

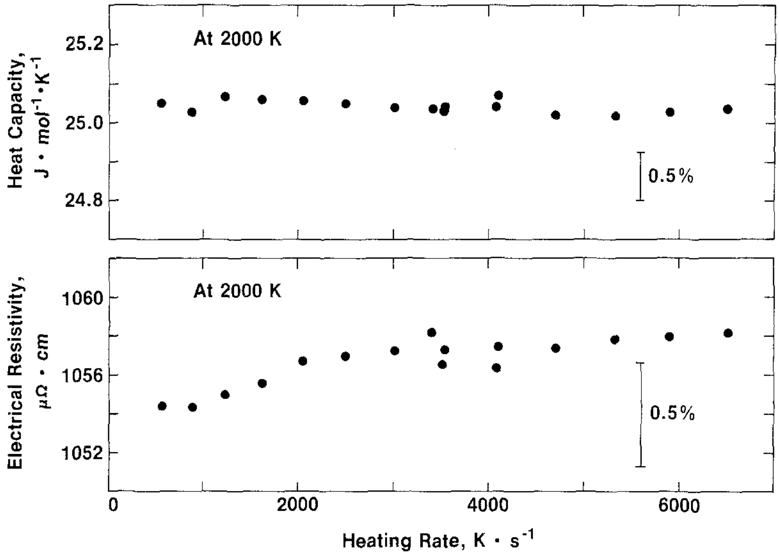

Fig. 4. Dependence of the measured heat capacity (at 2000 K) and electrical resistivity (at 2000 K) of POCO AXM-5Q1 graphite on the specimen heating rate.

blackbody hole at the specimen midpoint). This will tend to lower the measured value of any property (with a positive temperature coefficient) with respect to that determined under conditions of uniform temperature. This effect is evident in the results for electrical resistivity obtained at heating rates below about 2000 $K \cdot s^{-1}$ (see Fig. 4). On the basis of the values for heat capacity, electrical resistivity, and their temperature coefficients, a decrease of a similar magnitude is also expected in the measured heat capacity. For example, at 500 $K \cdot s^{-1}$ the observed value of resistivity is about 0.2% lower than those corresponding to heating rates above 2000 $K \cdot s^{-1}$. However, in the case of heat capacity, another (competing) effect arises. The presence of axial gradients will yield a temperature which will be lower than that measured in the absence of gradients. Since this difference in measured temperature will increase with temperature and since, in measurements by pulse-heating techniques, heat capacity is inversely proportional to dT/dt (rate of change of temperature with time), a second effect of a low heating rate will be to increase the measured value of heat capacity. Thus, errors in heat capacity arising from the above two effects are opposite in sign and, in the present work, appear to have about the same magnitude ($\sim$0.2% at 500 $K \cdot s^{-1}$).

The results in Fig. 4 for heat capacity tend to strengthen the basis for the calculation of radiative heat-loss corrections in measurements by pulse-heating techniques. As may be seen, there is no significant trend in heat capacity (at 2000 K) with decreasing heating rate even though the heat-loss correction (as a fraction of the input power) increases by almost one order of magnitude, from 6.5% at 6500 $K \cdot s^{-1}$ to 44% at 600 $K \cdot s^{-1}$. An uncertainty of, say, 5% in the heat-loss correction would yield a corresponding uncertainty in heat capacity of 0.3% at 6500 $K \cdot s^{-1}$, 0.5% at 3500 $K \cdot s^{-1}$, and 2.2% at 600 $K \cdot s^{-1}$. The results in Fig. 4 suggest that the uncertainty in heat-loss correction is considerably less than 5%.

4. DISCUSSION

Comparisons of our results for heat capacity and electrical resistivity with other literature data on graphites of the same grade are presented in Figs. 5 and 6, respectively. It should be noted that the specimen material for each investigation was obtained from the same lot of POCO AXM-5Q1 graphite (available at the OSRM at the NBS).

Below about 1200 K, there is good agreement between the heat capacity data of Taylor and Groot [5], obtained by scanning calorimetry, and the heat capacity values obtained by taking the first derivative of the relative enthalpy–temperature function determined by Ditmars [6] using a Bunsen ice calorimeter. These data appear to join smoothly with the heat-capacity results obtained in the present work. However, additional measurements at temperatures in the range 1200–1500 K would be valuable in accurately establishing the rapid change in slope of the heat capacity function in this temperature region.

As in the present work, considerable differences in the electrical resistivity of AXM-5Q1 graphite were also observed by Taylor and Groot [7] in measurements on three specimens, each with a different density. For comparison, the curves in Fig. 6 have been labeled with the room-temperature bulk density (in $g \cdot cm^{-3}$) of the graphite material used in each case. As expected, there is an inverse relationship between resistivity and density, that is, the highest resistivities are associated with the lowest densities, and vice versa. It may be seen that, in the temperature range common to the two investigations, the resistivity values obtained by Taylor and Groot exhibit a different slope, though the agreement among data for specimens of similar density is approximately within the combined experimental uncertainties ($\lesssim 3\%$). The agreement is less favorable at lower temperatures: the values obtained by Taylor and Groot at the resistivity minimum and at room temperature (293 K) for their specimen with density = 1.742 $g \cdot cm^{-3}$ are about 3 and 10% lower, respectively, than the present results for specimen 2 (density = 1.744 $g \cdot cm^{-3}$).

Prior to the present study, the electrical resistivity (at 293 K) had been measured by Hust [8] as a function of the position along sample rods 0045

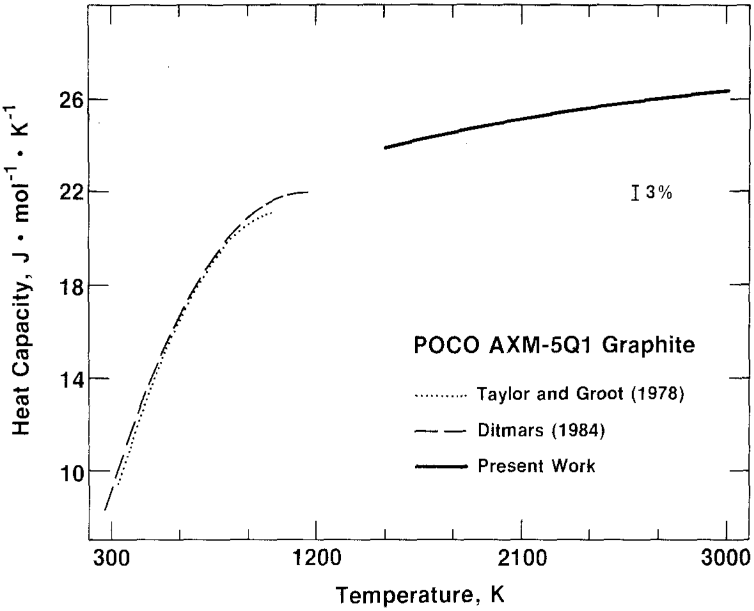

Fig. 5. Heat capacity of POCO AXM-5Q1 graphite: present work and data reported in the literature.

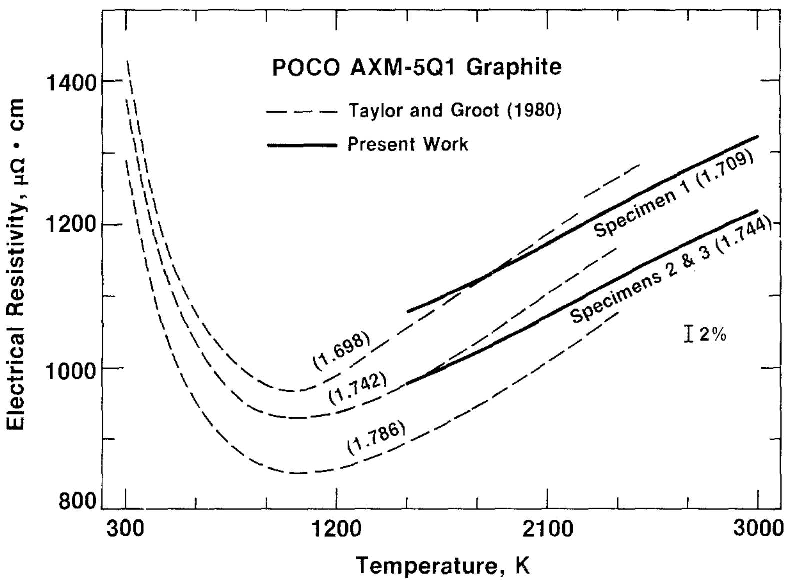

Fig. 6. Electrical resistivity of POCO AXM-5Q1 graphite: present work and data reported in the literature.

and 0046 (the source material for specimens 2 and 3 and specimen 1, respectively). Comparisons with Hust's data show that our results for specimens 2 and 3 are in good agreement ($\lesssim 1\%$) but our value for specimen 1 is nearly 6% higher. (This difference would be nearly accounted for if the portion of sample rod 0046 supplied by the OSRM had been taken from near "end 2" rather than from near "end 1," as was indicated.)

Although numerous investigations of the heat capacity of graphites of various grades are described in the literature, only a limited number of the measurements span a substantial portion of the present temperature range 1500–3000 K). A comparison of heat capacity data from the latter group of measurements and from the present work is given in Fig. 7.

Heat capacity data on POCO graphite and on pyrolytic graphite reported by Cezairliyan and Righini [9] are in good agreement with the present work. Their experiments yielded results for POCO grade DFP-2 that were not measurably different ($\sim 0.3\%$) from those for AXM-5Q[4] obtained earlier by Cezairliyan [10], using the same pulse-heating technique. As may be seen in Fig. 7, the (combined) heat capacity values for the two POCO grades is about 1% higher than the present results. Their

[4] AXM-5Q is a POCO grade of slightly lower purity than AXM-5Q1.

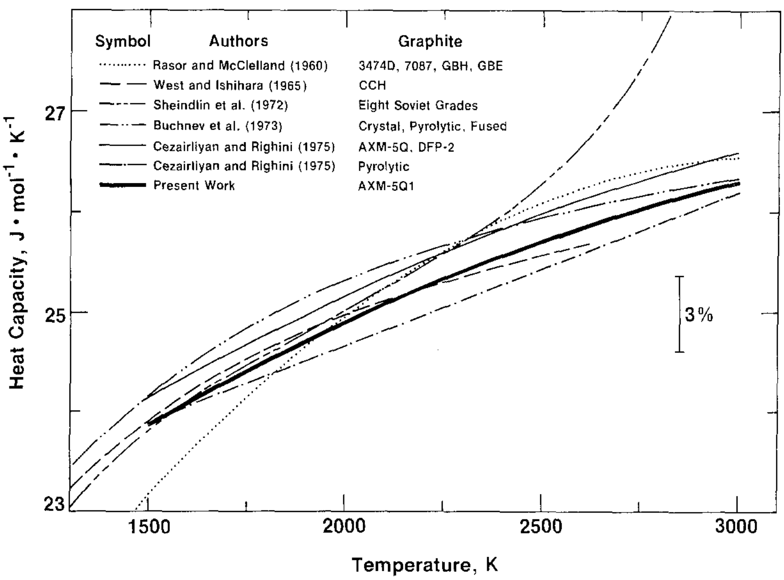

Fig. 7. Heat capacity of various grades and forms of graphite: present work and data reported in the literature.

results for pyrolytic graphite are about 1% (or less) lower than the present heat capacity values.

Several investigations have employed the drop method to measure high-temperature enthalpies of various graphites. The experimental study by Buchnev et al. [11] involved three forms of graphite: quasi-single crystal, pyrolytic, and glass (fused). Their enthalpy data for the different forms were reported to be the same within the measurement error (not given) and, therefore, were approximated by a single equation. The heat-capacity values which result are, on average, about 1% higher than the present values. Measurements by Sheindlin et al. [12] indicate that the enthalpies of eight Soviet grades do not differ by more than the reported experimental error ($\lesssim 2\%$). The resultant values for heat capacity are in good agreement with the present work in the range 1500 to 2300 K, but above 2300 K, they increase more rapidly with temperature, reaching a value at 3000 K which is 12% higher than the present value. The best agreement with the present measurements is provided by the enthalphy data of West and Ishihara [13] on grade CCH graphite, which yield heat capacity values within 0.5% of the present results throughout the overlapping temperature region (1500–2600 K).

Heat capacity data have also been reported by Rasor and McClelland [14] on four grades of graphite (3474D, 7087, GBH, GBE). Their results, which were obtained by pulse calorimetry, showed that differences with respect to graphite grade were within the experimental error ($\pm 5\%$). The dotted curve in Fig. 7, which represents a least-squares fit to their data (standard deviation = 4%), is in reasonable agreement ($\lesssim 3\%$) with the present results.

In summary, the present study suggests that differences among the literature data on the heat capacity of graphites at high temperatures are related more to systematic measurement errors than to any real dependence of heat capacity on the grade or form of graphite. Furthermore, the present work shows that the heat capacity of POCO graphite, grade AXM-5Q1, is well characterized at temperatures up to 3000 K, and therefore, this material would be a suitable high-temperature standard for heat capacity measurements. However, the large variations observed in electrical resistivity (as well as in thermal conductivity [7]) indicate that this material would make a less-than-ideal standard for the measurement of transport properties. As mentioned in a recent study [15], graphite can be a useful reference material, within limitations, only if the specimens are individually characterized by measurements of room-temperature density and electrical resistivity.

APPENDIX

Table AI. Experimental Results[a] for the Heat Capacity of POCO AXM-5Q1 Graphite

	Specimen 1		Specimen 2		Specimen 3	
T (K)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p (%)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p (%)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p (%)
Range I						
1500	23.85	−0.09	23.76	−0.46	23.64	−0.97
1550	24.00	0.07	23.93	−0.23	23.84	−0.60
1600	24.14	0.19	24.08	−0.06	24.04	−0.23
1650	24.25	0.19	24.22	0.07	24.22	0.07
1700	24.34	0.12	24.34	0.12	24.38	0.29
Range II						
1700	24.41	0.41	24.28	−0.13	24.20	−0.46
1750	24.52	0.43	24.41	−0.02	24.38	−0.14
1800	24.61	0.38	24.52	0.01	24.55	0.13
1850	24.70	0.34	24.62	0.01	24.71	0.38
1900	24.78	0.26	24.71	−0.02	24.86	0.59
Range III						
1900	24.76	0.18	24.66	−0.22	24.67	−0.18
1950	24.88	0.28	24.78	−0.12	24.82	0.04
2000	24.99	0.35	24.89	−0.06	24.97	0.27
2050	25.09	0.38	24.97	−0.10	25.10	0.42
2100	25.17	0.34	25.03	−0.22	25.22	0.54
Range IV						
2100	25.08	−0.02	25.04	−0.18	24.99	−0.37
2150	25.20	0.12	25.14	−0.12	25.13	−0.16
2200	25.31	0.22	25.24	−0.06	25.26	0.02
2250	25.41	0.29	25.32	−0.07	25.38	0.17
2300	25.49	0.29	25.38	−0.15	25.49	0.29
2350	25.55	0.22	25.43	−0.26	25.59	0.37
Range V						
2350	25.51	0.06	25.42	−0.29	25.34	−0.61
2400	25.58	0.04	25.49	−0.32	25.46	−0.43
2450	25.64	−0.02	25.57	−0.29	25.58	−0.25
2500	25.70	−0.06	25.64	−0.29	25.69	−0.10
2550	25.75	−0.13	25.72	−0.25	25.79	0.02
2600	25.80	−0.20	25.80	−0.20	25.89	0.15
2650	25.84	−0.29	25.89	−0.10	25.98	0.25
2700	25.89	−0.34	25.99	0.05	26.06	0.32
Range VI						
2700	25.88	−0.37	25.86	−0.45	25.89	−0.34
2750	25.92	−0.45	25.98	−0.22	26.01	−0.11
2800	25.96	−0.52	26.10	0.02	26.12	0.10
2850	25.99	−0.61	26.21	0.23	26.23	0.30
2900	26.03	−0.66	26.32	0.44	26.34	0.52
2950	26.07	−0.71	26.43	0.67	26.46	0.78
3000	26.13	−0.66	26.54	0.90	26.58	1.05

[a] ΔC_p is the percentage deviation of the individual results from the smooth function defined by Eq. (1).

Table AII. Experimental Results[a] for the Electrical Resistivity of POCO AXM-5Q1 Graphite

	Specimen 1		Specimen 2		Specimen 3	
T (K)	ρ ($\mu\Omega \cdot$cm)	$\Delta\rho$ (%)	ρ ($\mu\Omega \cdot$cm)	$\Delta\rho$ (%)	ρ ($\mu\Omega \cdot$cm)	$\Delta\rho$ (%)
Range I						
1500	1080.0	0.13	976.1	−0.09	980.3	0.34
1550	1086.0	0.05	982.6	−0.14	986.3	0.24
1600	1092.5	−0.01	989.4	−0.18	992.8	0.16
1650	1099.5	−0.05	996.5	−0.21	999.7	0.11
1700	1107.0	−0.06	1003.9	−0.23	1007.2	0.10
Range II						
1700	1107.0	−0.06	1004.5	−0.17	1007.1	0.09
1750	1114.9	−0.05	1012.3	−0.17	1014.8	0.08
1800	1122.9	−0.05	1020.2	−0.17	1022.8	0.08
1850	1131.1	−0.05	1028.4	−0.16	1031.1	0.10
1900	1139.4	−0.05	1036.9	−0.13	1039.7	0.14
Range III						
1900	1140.0	0.00	1036.6	−0.16	1039.2	0.09
1950	1148.5	0.00	1045.0	−0.15	1047.6	0.10
2000	1157.2	0.01	1053.6	−0.13	1056.2	0.12
2050	1166.1	0.04	1062.6	−0.08	1065.0	0.15
2100	1175.2	0.07	1071.8	−0.02	1073.9	0.18
Range IV						
2100	1174.7	0.02	1071.4	−0.05	1073.0	0.10
2150	1183.5	0.03	1080.1	−0.04	1081.7	0.10
2200	1192.4	0.03	1088.7	−0.05	1090.4	0.11
2250	1201.2	0.03	1097.3	−0.05	1099.1	0.11
2300	1210.0	0.03	1105.9	−0.06	1107.7	0.11
2350	1218.7	0.02	1114.5	−0.06	1116.2	0.09
Range V						
2350	1219.3	0.07	1114.8	−0.03	1116.9	0.16
2400	1227.9	0.05	1123.4	−0.03	1125.1	0.12
2450	1236.3	0.03	1131.8	−0.05	1133.4	0.09
2500	1244.7	0.01	1140.2	−0.06	1141.6	0.07
2550	1253.0	−0.01	1148.4	−0.08	1149.9	0.05
2600	1261.2	−0.03	1156.6	−0.09	1158.1	0.04
2650	1269.3	−0.04	1164.7	−0.10	1166.3	0.03
2700	1277.4	−0.05	1172.8	−0.11	1174.4	0.03
Range VI						
2700	1277.5	−0.04	1173.1	−0.08	1174.0	0.00
2750	1285.3	−0.05	1181.0	−0.09	1181.9	−0.01
2800	1293.0	−0.06	1188.9	−0.08	1189.9	0.00
2850	1300.8	−0.04	1196.8	−0.06	1197.8	0.02
2900	1308.6	−0.01	1204.6	−0.04	1205.6	0.04
2950	1316.3	0.04	1212.3	−0.01	1213.3	0.08
3000	1323.9	0.10	1220.0	0.04	1220.9	0.12

[a] $\Delta\rho$ is the percentage deviation of the individual results from the smooth functions defined by Eq. (2) for specimen 1 and Eq. (3) for specimens 2 and 3.

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