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Specific Heat Capacity and Electrical Resistivity of a Carbon-Carbon Composite in the Range 1500–3000 K by a Pulse Heating Method

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Measurements are described of specific heat capacity and electrical resistivity of a 2-2-3 T-50 carbon-carbon composite in the temperature range 1500–3000 K by a subsecond duration pulse heating technique. The results are represented by the relations

$$c_p = 1.691 + 2.598 \times 10^{-4} T - 2.691 \times 10^{-8} T^2$$
$$\rho = 733.0 + 6.594 \times 10^{-2} T$$

where c_p is in $J \cdot g^{-1} \cdot K^{-1}$, ρ is in $\mu\Omega \cdot cm$, and T is in K. Inaccuracy of specific heat capacity and electrical resistivity measurements is estimated to be not more than ±3%.

KEY WORDS: carbon-carbon; composites; dynamic measurements; electrical resistivity; graphite; heat capacity; high temperatures.

1. INTRODUCTION

Considerable effort has been devoted to the design, characterization, and testing of a variety of graphite-fiber reinforced composites for a number of applications, including the aerospace field, where materials with a high strength-to-weight ratio and stability at high temperatures are needed. However, as yet, little attention has been given to nonstructural aspects of these materials such as thermal and electrical properties. In this paper, we describe the measurement of specific heat capacity and electrical resistivity of

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a carbon-carbon composite by a pulse heating technique. This technique has been successfully used to study selected thermophysical properties of pure graphite [1], as well as several refractory metals and alloys [2].

The method involves measuring the specimen temperature, current through, and voltage across the specimen as it undergoes rapid resistive self-heating from room temperature to high temperatures (above 1500 K) in less than 1 sec. Temperature is measured at a rate of 1200 times per second by means of a high-speed photoelectric pyrometer [3]. Current through the specimen is determined from the measurement of the potential difference across a standard resistance placed in series with the specimen. Potential difference across the middle one-third of the specimen is measured between spring-loaded, knife-edge probes. These quantities are recorded digitally every 0.4 ms with a full-scale resolution of about 1 part in 8000.

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 properties, error analysis, etc., are given in earlier publications [4, 5].

2. MEASUREMENTS

2.1. Specimens

The specimen 2-2-3 T-50 carbon-carbon composite[2] consisted of a three-dimensional orthogonal weave of Thornel 50 graphite fibers impregnated with a matrix of coal tar pitch and graphitized at a processing temperature of 2750°C. The structure and physical properties characterizing the composite [6] are presented in Fig. 1 and Table I, respectively.

Two specimens were fabricated into the shape of tubes having the following nominal dimensions: length, 76 mm; outside diameter, 7.1 mm; and wall thickness, 0.9 mm. A small rectangular sighting hole (0.6 × 1 mm) was fabricated through the wall at the middle of each tube, thereby approximating a blackbody cavity for the pyrometric temperature measurements.

2.2. Procedure

The response of the high-speed pyrometer was optimized by dividing the temperature interval of the measurements (1500–3000 K) into six ranges. Prior to each experiment, the resistance in series with the specimen was adjusted in order to achieve the desired heating rate in a given temperature range. Each specimen was then pulse heated in a vacuum environment ($\sim 10^{-5}$ Torr) from room temperature successively through each temperature range,

[2]A rectangular sample from billet MT8812-1A was kindly furnished by the U.S. Air Force Materials Laboratory, Wright Patterson Air Force Base, Ohio.

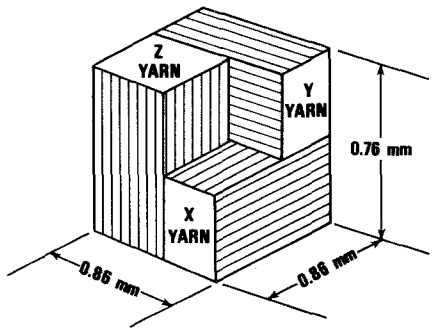

Fig. 1. Schematic diagram of a unit cell of the carbon-carbon composite, which consists of a three-dimensional orthogonal weave of yarns containing Thornel-50 fibers in a graphitic matrix.

beginning with the lowest range. The heating rates varied typically between 3800 and 4300 $K \cdot s^{-1}$. The duration of the current pulse ranged between 400 and 780 ms.

To study possible effects attributable to temperature in the vicinity of the processing temperature (2750°C) of the composite material, each specimen was "heat treated" by an additional nine heating pulses to 3000 K (2727°C) upon completion of the first series of experiments. The experiments in the successive temperature ranges were then repeated for each specimen. As will be described later, the heat treatment did not significantly affect the results for specific heat capacity, but it did alter the values obtained for electrical resistivity.

Optical checks performed on the specimen chamber window during the course of the experiments indicated the development of a light coating as a result of the ten consecutive pulse heatings to 3000 K. Weight measurements before and after the entire set of pulse experiments showed a 0.1% decrease in specimen mass.

Table I. Some Physical Properties of the 2-2-3 T-50 Carbon-Carbon Composite

	X yarn	Y yarn	Z yarn
Number of graphite fibers	2	2	3
Average spacing (mm)	0.86	0.86	0.76
Average spacing (in.)	0.034	0.034	0.030
Fiber volume fraction (%)	13	13	22
Matrix material	Coal tar pitch graphitized at 2750°C		
Matrix volume fraction (%)	52		
Bulk density[a] of composite ($g \cdot cm^{-3}$)	1.912		

[a] Determined by measurements on a cylindrical sample of specimen material.

Upon completion of all experiments, the high-speed pyrometer was calibrated with a tungsten filament reference lamp which, in turn, had been calibrated against the NBS Photoelectric Pyrometer by the Radiometric Physics Division at NBS. All temperatures reported in this work are based on the International Practical Temperature Scale of 1968 [7].

3. RESULTS

In all computations, the geometrical quantities were based on their room temperature (295 K) dimensions. The data on temperature, current, and voltage were fitted by the least squares method to a polynomial function of time for each quantity. The functions, in turn, were used to compute the values of specific heat capacity and electrical resistivity corresponding to each experiment; the results are given in Tables A1 and A2 of the Appendix. The final values for the properties were obtained by fitting the results in the Appendix to polynomials in temperature by the least squares method.

3.1. Specific Heat Capacity

The specific heat capacity of the carbon-carbon composite specimens was computed from data taken during the heating period. A correction for the radiative heat loss was based on the results obtained from the initial free cooling of the specimen, following the heating period, during the same experiments; typical values for this correction are given in Table II. The functions for specific heat capacity that represent the combined results of the two specimens for each series of measurements in the range 1500–3000 K are as follows:

For the first series (standard deviation = 0.7%),

$$c_p = 1.691 + 2.598 \times 10^{-4} T - 2.691 \times 10^{-8} T^2 \tag{1}$$

For the second series (standard deviation = 0.7%),

$$c_p = 1.829 + 1.278 \times 10^{-4} T + 3.876 \times 10^{-9} T^2 \tag{2}$$

Table II. Typical Values of the Radiative Heat Loss Correction for Pulse Heating Experiments on the Carbon-Carbon Composite

Temperature (K)	Radiative heat loss (% of input power)
1500	3
2000	7
2500	15
3000	33

where c_p is in J · g^{-1} · K^{-1} and T is in K. The smoothed values for specific heat capacity computed from Eqs. (1) and (2) are presented at 100 K intervals in Table III. It may be seen that the difference in specific heat capacity values obtained from the first and the second series of measurements is less than 1%.

3.2. Electrical Resistivity

The electrical resistivity of the carbon-carbon composite specimens was determined from the same experiments that were used to calculate specific heat capacity by means of the relation $\rho = RA/L$, where R is the measured resistance, A the cross-sectional area, and L the length of the specimen between the voltage probes. The cross-sectional area was obtained from a measurement of specimen mass and from the value of bulk density (1.912 g · cm^{-3}) determined by measurements on a cylindrical sample of specimen material. Unlike specific heat capacity, the results obtained for electrical resistivity was somewhat different for the two specimens, by about 3%. This difference may largely be attributed to anisotropies in the composite material and to differences in the fabrication of the specimens.

The functions for electrical resistivity that represent the combined results for the specimens obtained during the first series of experiments in the range 1500–3000 K are (standard deviation = 1.6%):

Table III. Smoothed Specific Heat Capacity for the Carbon-Carbon Composite

Temperature (K)	Specific heat capacity (J · g^{-1} · K^{-1})	
	First series	Second series[a]
1500	2.020	2.029
1600	2.038	2.043
1700	2.055	2.057
1800	2.071	2.072
1900	2.087	2.086
2000	2.103	2.100
2100	2.118	2.114
2200	2.132	2.129
2300	2.146	2.143
2400	2.160	2.158
2500	2.172	2.173
2600	2.185	2.187
2700	2.196	2.202
2800	2.207	2.217
2900	2.218	2.232
3000	2.228	2.247

[a]The data for the second series were taken after 10 successive (subsecond) pulse heatings from room temperature to 3000 K.

Table IV. Smoothed Electrical Resistivity for the Carbon-Carbon Composite

Temperature (K)	Electrical resistivity ($\mu\Omega \cdot$ cm)	
	First series	Second series[a]
1500	831.9	899.8
1600	838.5	908.6
1700	845.1	917.2
1800	851.7	925.3
1900	858.3	932.7
2000	864.9	939.3
2100	871.5	944.7
2200	878.1	948.8
2300	884.7	951.4
2400	891.3	952.3
2500	897.8	951.3
2600	904.4	948.1
2700	911.0	942.5
2800	917.6	934.4
2900	924.2	923.4
3000	930.8	—

[a]The data for the second series were taken after 10 successive (subsecond) pulse heatings from room temperature to 3000 K.

$$\rho = 733.0 + 6.594 \times 10^{-2} T \tag{3}$$

where ρ is in $\mu\Omega \cdot$ cm and T is in K. The second series of measurements, carried out after 10 successive pulse heatings to 3000 K, yielded a significant increase (as much as 5%) in electrical resistivity of the specimens at temperatures below 2900 K; above 2900 K, the resistivity values remained essentially the same as those obtained from the first series of experiments. The functions for electrical resistivity that represent the combined results for the two specimens obtained in the range 1500–2900 K during the second series are (standard deviation = 1.1%):

$$\rho = 878.7 - 1.396 \times 10^{-1} T + 1.549 \times 10^{-4} T^2 - 3.498 \times 10^{-8} T^3 \tag{4}$$

where ρ is in $\mu\Omega \cdot$ cm and T is in K. The smoothed values for electrical resistivity obtained from Eqs. (3) and (4) are given at 100 K intervals in Table IV.

Before the pulse experiments, the electrical resistivity of the two tubular specimens were measured at 295 K with a Kelvin bridge, yielding an average value of 1167 $\mu\Omega \cdot$ cm with an average absolute deviation of less than 1%.

4. ESTIMATE OF ERRORS

Estimates of errors in the measured and computed quantities lead to a value of ±3% as the maximum inaccuracy in both specific heat capacity and electrical resistivity reported in this work. Details regarding the estimates of errors and their combination in experiments with the present measurement system are given in a previous publication [5]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication.

It appears, on the basis of the following evidence, that a high degree of graphitization was achieved during fabrication of the composite material: (a) our results for specific heat capacity (previous section) seem to be insensitive to further heat treatment of the specimens; (b) our values for specific heat capacity of the carbon-carbon composite are in good agreement with the heat capacity data obtained earlier [1] for Poco graphite (see next section) using the same pulse heating system.

It is estimated that variations in density[3] through the billet of carbon-carbon composite used for specimen fabrication may contribute a maximum uncertainty of about 1.5% to the density value used in this work, which was determined from a "representative" cylindrical sample. The uncertainty in density, in turn, contributes maximum uncertainty of 1.5% to the cross-sectional area of each specimen, hence a 1.5% uncertainty in the electrical resistivity values.

Because of the high value of the hemispherical total emittance of the carbon-carbon composite, heat loss from the specimen due to thermal radiation constitutes a substantial fraction of the input power, especially at temperatures above 2500 K (see Table II). However, the scatter in the results for heat loss (~7% at 2000 K, decreasing to less than 1% at 3000 K) only contributes an uncertainty of about 0.5% to the heat capacity values throughout the temperature range of our measurements. Therefore, an accurate account of the magnitude of heat loss in the computation of heat capacity was possible.

Because of the inherent anisotropy of the composite material, nonuniformities in the electrical current and hence temperature gradients do exist within each unit cell (see Fig. 1) of the specimen during pulse heating. Our measurement technique, however, cannot resolve such gradients across a unit cell; indeed, our measurements of the experimental quantities (temperature, current, and voltage) are made on an "effective" specimen containing nearly 10^3 unit cells. In this sense, our experiments yield "average" values for the properties of material corresponding to the specimen bulk.

[3]The results of radiometric density measurements at 1 in. intervals throughout the composite were kindly supplied by the U.S. Air Force Materials Laboratory, Wright Patterson Air Force Base, Ohio.

5. DISCUSSION

There seems to be no other data in the literature for the thermophysical properties of carbon-carbon composites in the temperature range of our measurements. Consequently, we are using data for a similar material, Poco graphite, as the basis for comparison with our work. In Fig. 2, the specific heat capacity values obtained for the carbon-carbon composite during the first series of experiments are compared with the results for Poco graphite obtained by Cezairliyan and Righini [1] using the same pulse heating system. The difference in our results for the composite and those of Poco graphite is smaller than 1%, which is less than the maximum estimated inaccuracy in our measurements (~3%). Similar agreement exists for the specific heat capacity values obtained during our second series of measurements. This agreement suggests that the matrix, in which the graphite fibers are embedded, is essentially graphitic.

The effect of multiple pulse heatings to 3000 K on electrical resistivity is illustrated in Fig. 3. It may be seen that resistivity increases with each successive pulse heating, although the magnitude of this increase decreases rapidly with additional pulse heatings. An examination of the specimen surface upon completion of the experiments revealed the existence of microscopic cracks between the yarns containing the graphite fibers and the graphitic matrix. The separations of the yarn from the surrounding matrix undoubtedly affect the transport properties of the composite in such a way as to increase its electrical resistance. Differences in thermal expansion between fibers and matrix are probably responsible for development of the cracks.

In Fig. 4, we present our values for electrical resistivity of the composite along with results for two different types of Poco graphite reported earlier [1].

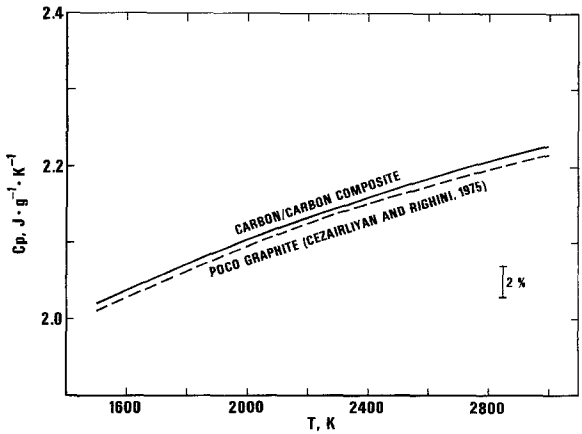

Fig. 2. A comparison of specific heat capacity values for the carbon-carbon composite with those obtained by Cezairliyan and Righini [1] for Poco graphite.

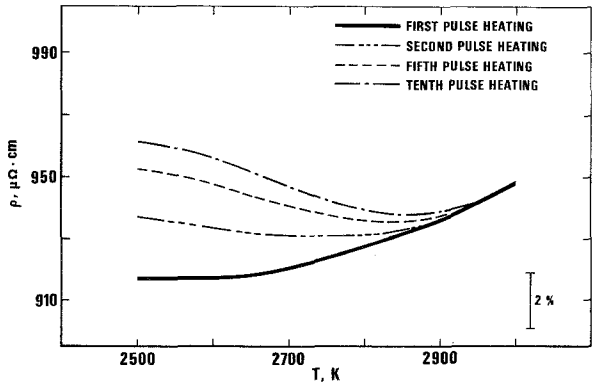

Fig. 3. Illustration of the effect on electrical resistivity for the carbon-carbon composite of successive multiple pulse heatings to 3000 K.

In view of the difference in electrical resistivity (~7%) between different Poco graphite specimens [1], the even larger difference observed between the results for the composite and the Poco graphite may be expected. However, the data for the composite and the Poco graphite have approximately the same temperature dependence: (a) at temperatures near room temperature, the electrical resistivity of the composite exhibits a large negative temperature coefficient (see Fig. 5), as is the case of Poco graphite [8]; (b) after passing through a minimum at an intermediate temperature, the resistivity values for both composite (first series) and Poco graphite increase approximately linearly with temperature as shown in Fig. 4. The variation of

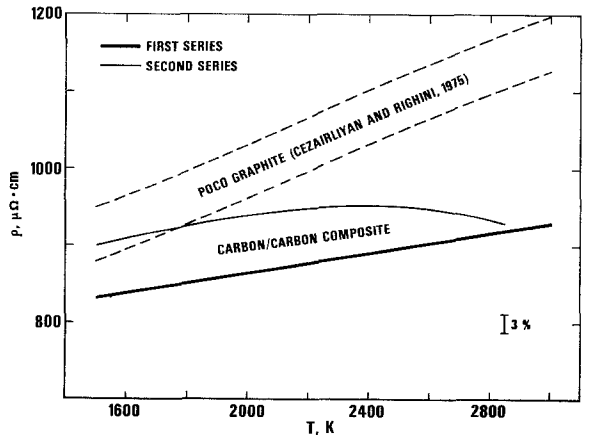

Fig. 4. A comparison of electrical resistivity values for the carbon-carbon composite with those obtained by Cezairliyan and Righini [1] for two different types of Poco graphite: AXM-5*Q* (upper dashed line), DFP-2 (lower dashed line).

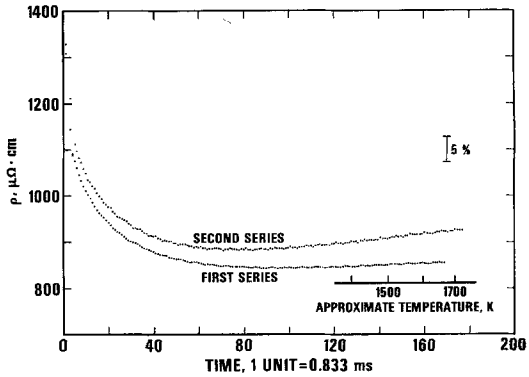

Fig. 5. Electrical resistivity of the carbon-carbon composite as a function of time for experiments in which the specimen is pulse heated from room temperature to approximately 1700 K.

electrical resistivity with changing temperature in Poco graphite can be explained in terms of a semicontinuum model [8] in which the predominant influence on electrical resistivity arises from crystal boundary scattering at low temperatures (<700 K) and from phonon-phonon scattering a high temperatures. The same mechanisms appear to be involved in determining the electrical resistivity of the carbon-carbon composite.

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APPENDIX: TABLES A1 AND A2

Table A1. Experimental Results on Specific Heat Capacity of the Carbon-Carbon Composite

Pyrometer range	T (K)	First series				Second series[a]			
		Specimen 1		Specimen 2		Specimen 1		Specimen 2	
		c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[b] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[b] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[c] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[c] (%)
I	1500	1.999	−1.1	2.005	−0.8	2.017	−0.6	2.004	−1.3
	1550	2.021	−0.4	2.027	−0.1	2.032	−0.2	2.028	−0.4
	1600	2.041	+0.2	2.049	+0.5	2.048	+0.2	2.049	+0.3
	1650	2.061	+0.7	2.070	+1.1	2.064	+0.7	2.068	+0.9
II	1650	2.027	−1.0	2.037	−0.5	2.035	−0.8	2.035	−0.8
	1700	2.048	−0.3	2.052	−0.1	2.052	−0.3	2.053	−0.2
	1750	2.070	+0.3	2.069	+0.3	2.071	+0.3	2.071	+0.3
	1800	2.091	+0.9	2.086	+0.7	2.087	+0.7	2.089	+0.8
III	1800	2.062	−0.5	2.062	−0.5	2.061	−0.5	2.065	−0.3
	1850	2.078	−0.1	2.078	−0.1	2.077	−0.1	2.082	+0.2
	1900	2.094	+0.3	2.095	+0.4	2.094	+0.4	2.097	+0.5
	1950	2.108	+0.6	2.110	+0.7	2.110	+0.8	2.113	+1.0
	2000	2.122	+0.9	2.124	+1.0	2.125	+1.2	2.127	+1.3

Table A1. Continued.

Pyrometer range	T (K)	First series				Second series[a]			
		Specimen 1		Specimen 2		Specimen 1		Specimen 2	
		c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[b] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[b] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[c] (%)	c_p ($J \cdot g^{-1} \cdot K^{-1}$)	Δc_p[c] (%)
IV	2000	2.091	−0.6	2.096	−0.3	2.084	−0.8	2.083	−0.8
	2050	2.106	−0.2	2.107	−0.2	2.099	−0.4	2.100	−0.3
	2100	2.119	+0.1	2.118	0	2.114	−0.1	2.115	+0.1
	2150	2.131	+0.3	2.130	+0.2	2.129	+0.4	2.129	+0.4
	2200	2.143	+0.5	2.143	+0.5	2.143	+0.7	2.142	+0.6
	2250	2.154	+0.7	2.158	+0.9	2.157	+1.0	2.154	+0.8
V	2200	2.114	−0.9	2.092	−1.9	2.108	−1.0	2.102	−1.3
	2300	2.137	−0.4	2.132	−0.7	2.132	−0.5	2.130	−0.6
	2400	2.159	−0.1	2.151	−0.4	2.151	−0.3	2.154	−0.2
	2500	2.181	+0.4	2.171	−0.1	2.172	−0.1	2.175	+0.1
	2600	2.203	+0.8	2.192	+0.3	2.197	+0.4	2.195	+0.3
VI	2500	2.160	−0.6	2.148	−1.1	2.152	−1.0	2.143	−1.4
	2600	2.181	−0.2	2.165	−0.9	2.173	−0.7	2.173	−0.7
	2700	2.199	+0.1	2.179	−0.8	2.193	−0.4	2.195	−0.3
	2800	2.218	+0.5	2.193	−0.7	2.213	−0.2	2.214	−0.1
	2900	2.234	+0.7	2.207	−0.5	2.233	+0.1	2.235	+0.1
	3000	2.252	+1.0	2.227	−0.1	2.259	+0.5	2.272	+1.1

[a] Second series data were obtained after 10 successive (subsecond) pulse heatings from room temperature to 3000 K.

[b] Δc_p is the percentage deviation of the individual results for specimens 1 and 2 (obtained during the first series) from the smooth function defined by Eq. (1).

[c] Δc_p is the percentage deviation of the individual results for specimens 1 and 2 (obtained during the second series) from the smooth function defined by Eq. (2).

Table A2. Experimental Results on Electrical Resistivity of the Carbon-Carbon Composite

Pyrometer range	T (K)	First series				Second series[a]			
		Specimen 1		Specimen 2		Specimen 1		Specimen 2	
		ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[b] (%)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[b] (%)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[c] (%)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[c] (%)
I	1500	849.8	+2.1	815.0	−2.1	910.3	+1.2	889.7	−1.1
	1550	851.1	+1.9	817.8	−2.1	914.7	+1.2	894.2	−1.1
	1600	852.5	+1.6	820.7	−2.2	918.8	+1.1	898.3	−1.1
	1650	854.0	+1.4	823.7	−2.2	922.4	+1.0	902.1	−1.2
II	1650	855.0	+1.5	831.0	−1.3	922.7	+1.1	904.1	−1.0
	1700	856.4	+1.3	832.3	−1.5	925.9	+1.0	907.3	−1.1
	1750	858.5	+1.2	834.3	−1.7	929.3	+0.9	910.6	−1.2
	1800	861.4	+1.1	837.1	−1.7	932.8	+0.8	914.1	−1.2
III	1800	868.0	+1.9	842.8	−1.0	936.1	+1.2	918.0	−0.8
	1850	869.0	+1.6	844.0	−1.3	938.8	+1.1	920.3	−0.9
	1900	870.7	+1.4	845.7	−1.5	941.6	+1.0	922.8	−1.1
	1950	873.1	+1.3	848.1	−1.6	944.4	+0.9	925.3	−1.1
	2000	876.2	+1.3	851.1	−1.6	947.4	+0.9	928.0	−1.2

Table A2. Continued.

Pyrometer range	T (K)	First series, Specimen 1 ρ ($\mu\Omega \cdot$ cm)	First series, Specimen 1 $\Delta\rho$[b] (%)	First series, Specimen 2 ρ ($\mu\Omega \cdot$ cm)	First series, Specimen 2 $\Delta\rho$[b] (%)	Second series[a], Specimen 1 ρ ($\mu\Omega \cdot$ cm)	Second series[a], Specimen 1 $\Delta\rho$[c] (%)	Second series[a], Specimen 2 ρ ($\mu\Omega \cdot$ cm)	Second series[a], Specimen 2 $\Delta\rho$[c] (%)
IV	2000	880.9	+1.8	854.7	−1.2	949.9	+1.1	930.9	−0.9
	2050	882.2	+1.6	855.8	−1.4	951.3	+1.0	932.1	−1.1
	2100	884.1	+1.4	857.9	−1.6	953.2	+0.9	933.6	−1.2
	2150	886.6	+1.3	860.6	−1.6	955.2	+0.9	935.3	−1.2
	2200	889.5	+1.3	863.7	−1.7	957.2	+0.9	937.0	−1.2
	2250	892.6	+1.3	866.9	−1.7	958.9	+0.9	938.5	−1.2
V	2200	899.8	+2.4	873.9	−0.5	963.7	+1.6	944.0	−0.5
	2300	899.3	+1.6	874.3	−1.2	962.7	+1.2	943.1	−0.9
	2400	902.2	+1.2	877.6	−1.6	962.4	+1.1	941.9	−1.1
	2500	907.5	+1.1	883.0	−1.7	961.5	+1.1	939.8	−1.2
	2600	913.7	+1.0	889.4	−1.7	958.3	+1.1	935.7	−1.3
VI	2500	917.7	+2.2	891.2	−0.7	964.2	+1.4	943.8	−0.8
	2600	917.5	+1.4	891.7	−1.4	959.4	+1.2	937.6	−1.1
	2700	921.1	+1.1	895.4	−1.7	950.0	+0.8	927.9	−1.5
	2800	927.8	+1.1	901.9	−1.7	941.0	+0.7	918.8	−1.6
	2900	936.5	+1.3	910.2	−1.5	938.4	+1.6	915.4	−0.8
	3000	945.9	+1.6	919.2	−1.3	950.9	—	924.9	—

[a] Second series data were obtained after 10 successive (subsecond) pulse heatings from room temperature to 3000 K.

[b] $\Delta\rho$ is the percentage deviation of the individual results for specimens 1 and 2 (obtained during the first series) from the smooth function defined by Eq. (3).

[c] $\Delta\rho$ is the percentage deviation of the individual results for specimens 1 and 2 (obtained during the second series) from the smooth function defined by Eq. (4).