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Heat Capacity and Electrical Resistivity of Palladium in the Range 1400 to 1800 K by a Pulse Heating Method

A. P. Miiller[1] and A. Cezairliyan[1]

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Measurements of heat capacity and electrical resistivity of palladium in the temperature range 1400–1800 K by a subsecond duration pulse heating technique are described. The results are expressed by the relations:

$$C_p = 32.19 - 5.966 \times 10^{-3}T + 4.440 \times 10^{-6}T^2$$

$$\rho = 15.42 + 1.840 \times 10^{-2}T$$

where C_p is in J · mol^{-1} · K^{-1}, ρ in $\mu\Omega$ · cm, and T in K. Estimated maximum inaccuracies of the measured properties are: 3% for heat capacity and 1% for electrical resistivity.

KEY WORDS: electrical resistivity; heat capacity; high temperature; palladium; pulse method.

1. INTRODUCTION

There is a paucity of data in the literature for some of the thermophysical properties of palladium at high temperatures, particularly for heat capacity and electrical resistivity. The few existing investigations have utilized techniques that expose the specimens to high temperatures for relatively long periods of time (minutes to hours), creating problems that result from increased heat loss, chemical reactions, evaporation, etc. Specimen evaporation, in particular, is enhanced by the relatively large vapor pressure of

[1]Thermophysics Division, National Bureau of Standards, Washington, D.C. 20234, USA.

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palladium near its melting point. In order to reduce the effects of these problems, we have used a subsecond-duration pulse heating technique to measure the heat capacity and electrical resistivity of palladium at temperatures between 1400 and 1800 K. This technique has been used successfully to measure selected thermophysical properties of a number of refractory materials [1].

The method involves measuring the specimen temperature and the current through and voltage across the specimen as it undergoes rapid resistive self-heating from room temperature to high temperatures in less than 1 s. The temperature is measured by means of a high-speed photoelectric pyrometer [2]. The current through the specimen is determined by measuring the voltage across a standard resistance in series with the specimen. The voltage across the middle one-third of the specimen is measured between spring-loaded knife edge probes. The 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 and other pertinent information, such as formulation of relations for properties, error analysis, etc., are given in earlier publications [3, 4].

2. MEASUREMENTS

Emission spectrographic analyses by the manufacturer (of typical material nominally $99.95^{+}\%$ pure) yielded the following impurities (in ppm by mass): Si, 150; Mg, 55; Fe, 45; Al, 20; Zr, 15; Pt, Au, 10 each; Cu, less than 10. The material, supplied in the form of cylindrical rods, was fabricated into three tubular specimens by an electroerosion technique. The dimensions of the tubes were nominally: length, 75 mm; outside diameter, 6.4 mm; wall thickness, 0.5 mm. A rectangular sighting hole (0.5×1 mm) was fabricated through the wall at the middle of each tube, thereby approximating a blackbody cavity for the pyrometric temperature measurements.

The response of the high-speed pyrometer [2] was optimized by dividing the temperature interval of the measurements (1400–1800 K) into three ranges. One experiment per specimen was performed in each temperature range. Prior to each experiment, we adjusted a resistance in series with the specimen in order to achieve the desired heating rate in a given temperature range. The specimen was then heated in an argon environment at about 0.2 MPa (about 2 atm) from room temperature to the desired temperature by the passage of an electrical current pulse through it. Heating rates ranged typically between 2700 and 3900 $K \cdot s^{-1}$. The duration of the current pulse, which varied between 500 and 600 ms, determined the maximum specimen temperature achieved in a given experiment. Upon completion of the experiments, we calibrated the high-speed pyrometer using a tungsten filament

Table I. Smoothed Heat Capacity and Electrical Resistivity of Palladium

T (K)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ρ ($\mu\Omega \cdot cm$)
1400	32.54	41.18
1450	32.87	42.10
1500	33.23	43.02
1550	33.61	43.94
1600	34.01	44.86
1650	34.43	45.78
1700	34.88	46.70
1750	35.35	47.62
1800	35.84	48.54

reference lamp which, in turn, had been calibrated against the NBS "Temperature Standard." All temperatures reported in this work are based on the International Practical Temperature Scale of 1968 [5].

3. RESULTS

The data on voltage, current, and temperature within each temperature range were fitted to second-degree polynomial functions for each quantity in terms of time by the least-squares method. The functions were then used to compute[2] the values of 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 results in the Appendix to polynomials in temperature by the least-squares method. The results are presented at 50 K temperature intervals in Table I.

3.1. Heat Capacity

We computed the heat capacity using the data taken during the heating period. Corrections were made for the radiative heat loss from the specimen which, based on the data taken during the initial cooling period of the specimen, amounted to less than 2% at 1400 K and less than 3% at 1800 K. The function that represents the results for heat capacity (standard deviation = 0.8%) in the temperature range from 1400 to 1800 K is

$$C_p = 32.19 - 5.966 \times 10^{-3}T + 4.440 \times 10^{-6}T^2 \quad (1)$$

[2]In all computations, the geometrical quantities are based on their room temperature (298 K) dimensions.

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 palladium was taken as 106.4.

3.2. Electrical Resistivity

The electrical restivity of the specimens 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 palladium ($12.02 \text{ g} \cdot \text{cm}^{-3}$) and a measurement of the specimen weight. The function that represents the results for electrical resistivity (standard deviation = 0.2%) in the temperature range 1400 to 1800 K is

$$\rho = 15.42 + 1.840 \times 10^{-2} T \qquad (2)$$

where ρ is in $\mu\Omega \cdot \text{cm}$ and T is in K. Prior to the pulse experiments, a Kelvin bridge was used to measure the electrical resistivity of the three tubular specimens at "room temperature," yielding an average value of $11.1 \ \mu\Omega \cdot \text{cm}$ at 288 K, with an average absolute deviation of 0.3% and a maximum absolute deviation of 0.4%.

3.3. Estimate of Errors

The methods of estimating errors in the measured and computed quantities have been discussed at length in an earlier publication [4]. Items in the error analysis were recomputed whenever present conditions differed from those in the earlier publication. The maximum errors in the reported values are estimated to be 3% in the heat capacity and 1% in the electrical resistivity.

4. DISCUSSION

The heat capacity and electrical resistivity of palladium measured in this work are compared graphically with those reported in the literature in Figs. 1 and 2, respectively.

The heat capacity data of Jaeger and Veenstra [6] were obtained by drop calorimetry; this work revised earlier less accurate data by Jaeger and coworkers (see Ref. [7]), using the same technique. The recent data obtained by Voolmer and Kohlhaas [8] using adiabatic calorimetry exhibit a rather different trend with changing temperature: their heat capacity values rise more rapidly with increasing temperature, ranging from 2% lower than the results of Jaeger and Veenstra at 700 K to about 5% higher at 1800 K. The results of these studies are in agreement with those of the present work within 4%. The data of Vollmer and Kohlhaas are about 1% or less higher than our

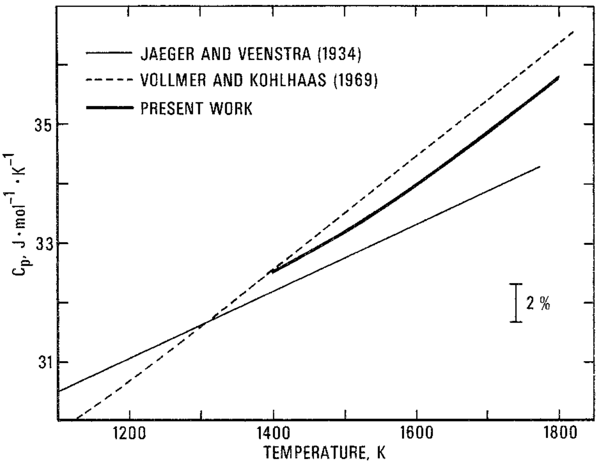

Fig. 1. Heat capacity of palladium: present work and data reported in the literature.

results, whereas the results of Jaeger and Veenstra range from approximately 1% lower at 1400 K to about 4% lower at 1800 K. The trend of the latter data at temperatures below 1400 K appears to be in better agreement with our work than the trend exhibited by the data of Vollmer and Kohlhaas.

The only values in the literature for electrical resistivity of palladium above 1000 K appear to be those reported by Landensperger and Stark [9] for the range 1200–1500 K, and the results of Laubitz and Matsumura [10] for the range 90–1300 K. A linear extrapolation of the latter results to 1400 K

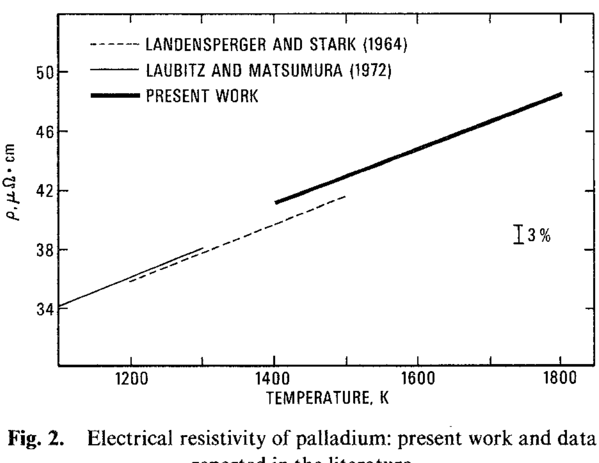

Fig. 2. Electrical resistivity of palladium: present work and data reported in the literature.

yields a value which is about 1 $\mu\Omega \cdot$ cm (or 2.5%) lower than the value obtained in the present work, whereas the value reported by Landensperger and Stark at 1400 K is nearly 3% lower than our result. These differences lie within combined experimental (and extrapolation) errors. A comparison of the "room temperature" resistivity values obtained by Laubitz and Matsumura and by ourselves shows that their results are again about 1 $\mu\Omega \cdot$ cm lower than our value, even though in both investigations, the low and the high temperature data were obtained by independent measurement techniques. This suggests that the lower resistivities obtained by Laubitz and Matsumura might have been partially due to the higher purity of their specimens, 99.99%, compared with 99.95% pure specimen material used in our experiments. Landensperger and Stark did not report the purity of their specimen material.

APPENDIX: TABLES A1 AND A2

Table A1. Experimental Results for the Heat Capacity of Palladium

	Specimen 1		Specimen 2		Specimen 3	
T (K)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p[a] (%)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p[a] (%)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ΔC_p[a] (%)
Range I						
1400	32.36	−0.6	32.72	+0.6	32.57	+0.1
1450	32.70	−0.5	33.03	+0.5	33.04	+0.5
1500	33.02	−0.6	33.32	+0.3	33.48	+0.7
1550	33.34	−0.8	33.59	−0.1	33.88	+0.8
Range II						
1500	32.98	−0.8	33.17	−0.2	33.16	−0.2
1550	33.39	−0.7	33.61	+0.1	33.72	+0.3
1600	33.80	−0.6	34.08	+0.2	34.27	+0.8
1650	34.19	−0.7	34.57	+0.4	34.83	+1.1
1700	34.57	−0.9	35.09	+0.6	35.38	+1.4
Range III						
1650	33.98	−1.3	34.39	−0.1	34.60	+0.5
1700	34.50	−1.1	34.77	−0.3	35.20	+0.9
1750	35.02	−0.9	35.14	−0.6	35.81	+1.3
1800	35.53	−0.9	35.50	−0.9	36.41	+1.6

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

Table A2. Experimental Results for the Electrical Resistivity of Palladium

	Specimen 1		Specimen 2		Specimen 3	
T (K)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[a] (%)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[a] (%)	ρ ($\mu\Omega \cdot$ cm)	$\Delta\rho$[a] (%)
Range I						
1400	41.25	+0.2	41.05	−0.3	41.18	−0.1
1450	42.18	+0.2	41.98	−0.3	42.15	+0.1
1500	43.12	+0.2	42.91	−0.3	43.09	+0.2
1550	44.05	+0.2	43.84	−0.2	44.01	+0.2
Range II						
1500	43.11	+0.2	42.90	−0.3	43.07	+0.1
1550	44.02	+0.2	43.81	−0.3	43.98	+0.1
1600	44.94	+0.2	44.72	−0.3	44.89	+0.1
1650	45.87	+0.2	45.64	−0.3	45.81	+0.1
1700	46.80	+0.2	46.56	−0.3	46.75	+0.1
Range III						
1650	45.87	+0.2	45.64	−0.3	45.82	+0.1
1700	46.79	+0.2	46.55	−0.3	46.73	+0.1
1750	47.72	+0.2	47.47	−0.3	47.66	+0.1
1800	48.66	+0.2	48.39	−0.3	48.61	+0.1

[a] $\Delta\rho$ is the percentage deviation of the individual results from the smooth function defined by Eq. (2).

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