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Simultaneous Measurements of Heat Capacity, Electrical Resistivity and Hemispherical Total Emittance by a Pulse Heating Technique: Zirconium, 1500 to 2100 K*

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Simultaneous measurements of heat capacity, electrical resistivity and hemispherical total emittance of zirconium in the temperature range 1500 to 2100 K by a subsecond duration, pulse heating technique are described. The results are expressed by the relations:

$$C_p = 36.65 - 1.435 \times 10^{-2}\,T + 6.624 \times 10^{-6}T^2$$

$$\rho = 87.95 + 1.946 \times 10^{-2}T$$

$$\epsilon = 0.2031 + 6.362 \times 10^{-5}T$$

where C_p is in $\mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, ρ is in $10^{-8}\ \Omega \cdot \mathrm{m}$, and T is in K. Estimated inaccuracies of the measured properties are: 3 percent for heat capacity, 2 percent for electrical resistivity and 5 percent for hemispherical total emittance.

Key words: Electrical resistivity; emittance; heat capacity; high-speed measurements; high temperature; thermodynamics; thermophysical properties; zirconium.

1. Introduction

In this paper, application of a pulse heating technique to the simultaneous measurements of heat capacity, electrical resistivity and hemispherical total emittance of zirconium in the temperature range 1500 to 2100 K is described.

The method is based on rapid resistive self-heating of the specimen from room temperature to high temperatures (above 1500 K) in less than one second by the passage of an electrical current pulse through it; and on measuring, with millisecond resolution, such experimental quantities as current through the specimen, potential drop across the specimen, and specimen temperature. 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 [1, 2].[1]

In the following sections of this paper a tabular format is adopted in presenting information on the specimen, measurements, system characteristics, results and errors.

2. Measurements

The details regarding the zirconium specimens used in the present measurements are given in table 1. A summary of the measurement technique and the operational characteristics of the system is given in table 2. The polynomial functions (obtained by the least squares method) that represent the experimental results are given in table 3. The values of properties at 100 degree temperature intervals computed using the functions are given in table 4. The experimental results are presented in the appendix. Each number tabulated in the appendix represents results from over fifty original data points. An estimate of errors in the measured and computed quantities is given in table 5. All values reported in this paper are based on the International Practical Temperature Scale of 1968 [3]. In all computations, the geometrical quantities are based on their room temperature (298 K) dimensions.

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[1] Figures in brackets indicate the literature references at the end of this paper.

TABLE 1. *Specimen information*

No.	Item	Unit	Explanation
1	Substance		Zirconium (polycrystalline).
2	Source[a]		Materials Research Corporation.
3	Purity		99.98%.
4	Impurities		Listed in table 1a.
5	Geometry		Tube made from rod by electro-erosion.
6	Dimensions (nominal)		
	total length	mm	76.2
	effective[b] length	mm	25.4
	outside diameter	mm	6.3
	wall thickness	mm	0.5
	blackbody hole	mm	0.5 × 1 (rectangular).
7	Weight		
	total weight	g	4.312
	effective[b] weight	g	1.425
8	Characteristics		
	atomic weight [4]		91.22
	density[c]	$g \cdot cm^{-3}$	6.53
	resistivity at 293 K	$10^{-8} \Omega \cdot m$	42.8

[a] The supplier is identified in this paper in order to adequately characterize the specimen. Such an identification does not imply recommendation or endorsement by the National Bureau of Standards.

[b] Effective refers to the portion of the specimen between the voltage probes.

[c] Measured value in the present work.

TABLE 1a. *Impurities in the specimen*[a]

(according to the manufacturer's analysis)

Element	C	H	O	N	Al	Fe	Hf	Ni	Si	Ti
ppm	6	3.3	125	2.1	3	30	40	1.5	1.5	1

[a] The total amount of all other detected elements is less than 6 ppm, each element being below 1 ppm limit.

TABLE 2. *Measurement technique and system characteristics*

No.	Item	Unit	Explanation and data
1	General technique		Pulse heating (subsecond).
2	Voltage measurement		Across tungsten knife-edge probes.
3	Current measurement		Across standard resistor (0.001 Ω) in series with the specimen.
4	Temperature measurement		High-speed photoelectric pyrometer [5].
5	Specimen environment		Vacuum ~ 1.3×10^{-3} $N \cdot m^{-2}$ (~ 10^{-5} torr).
6	Power source		Battery bank (14 series-connected 2V batteries, capacity 1100 A·h each).
7	Recording		Digital data acquisition system.
8	Signal resolution		~ 0.01 percent (at full scale).
9	Time resolution	ms	0.4
10	Data processing		Time-sharing computer.
11	Number of specimens		3
12	Number of experiments		12
13	Temperature range	K	1500–2100
14	Temperature subranges	K	I (1450–1680) II (1680–1900) III (1810–2050) IV (1840–2110)
15	Experiment duration	ms	550–680
16	Current pulse length	ms	350–480
17	Imparted power	W	1700–3100
18	Current	A	730–840
19	Rate of current change	$A \cdot ms^{-1}$	0.08–0.13
20	Heating rate	$K \cdot ms^{-1}$	2.9–4.8
21	Cooling rate	$K \cdot ms^{-1}$	0.08–0.3
22	Radiative heat loss (Percent of input power)		2 percent at 1500 K 10 percent at 2100 K

TABLE 3. *Functional representation of results on zirconium*

Heat capacity ($J \cdot mol^{-1} \cdot K^{-1}$)	Resistivity ($10^{-8} \Omega \cdot m$)	Hemispherical total emittance
$C_p = a + bT + cT^2$	$\rho = a + bT$	$\epsilon = a + bT$
$a = 36.65$ $b = -1.435 \times 10^{-2}$ $c = 6.624 \times 10^{-6}$	$a = 87.95$ $b = 1.946 \times 10^{-2}$	$a = 0.2031$ $b = 6.362 \times 10^{-5}$
$1500\ K < T < 2100\ K$ $\sigma^a = 0.7\%$	$1500\ K < T < 2100\ K$ $\sigma^a = 0.6\%$	$1650\ K < T < 2050\ K$ $\sigma^a = 0.9\%$

[a]Standard deviation as computed from the difference between the value of an experimental result (as tabulated in the appendix) and that from the smooth functions reported above.

TABLE 4. *Results on properties of zirconium*

T (K)	C_p ($J \cdot mol^{-1} \cdot K^{-1}$)	ρ ($10^{-8} \Omega \cdot m$)	ϵ
1500	30.03	117.14	[a] 0.299
1600	30.65	119.09	[a] .305
1700	31.40	121.03	.311
1800	32.28	122.98	.318
1900	33.30	124.92	.324
2000	34.45	126.87	.330
2100	35.73	128.82	[a] .337

[a] Extrapolated values.

TABLE 5. *Error analysis (at 2000 K)*

Quantity	Imprecision [a]	Inaccuracy [b]
Temperature	0.5 K	4 K
Voltage	0.03%	0.1%
Current	0.03%	0.1%
Heat capacity	0.7%	3%
Electrical resistivity	0.6%	2%
Hemispherical total emittance	0.9%	5%

[a] Imprecision refers to the standard deviation of a quantity as computed from the difference between the value of the quantity and that from the smooth function obtained by the least squares method. The quantities in the case of temperature, voltage, and current are the individual points measured in a single experiment, and in the case of heat capacity, electrical resistivity, and hemispherical total emittance are the results from all experiments as tabulated in the appendix.

[b] Inaccuracy refers to the estimated total error (random and systematic).

3. Discussion

The differences in the measured properties for the three specimens were within the measurement resolution for the properties, and the final smoothed results (represented by the equations in table 3 and tabulations in table 4) were obtained from the combined data for the three specimens. The heat capacity, electrical

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resistivity and hemispherical total emittance of zirconium measured in this work are presented and compared graphically with those reported in the literature in figures 1, 2, and 3, respectively. The present results are for temperatures up to 2100 K, which is approximately 30 K below the melting point of zirconium.

The heat capacity results of this work are approximately 1 percent lower than those of Skinner [6] in the overlapping temperature region. Extrapolation of the results of this work to lower temperatures (1200–1400 K) yields values which are 3–6 percent lower than those reported by Coughlin and King [7]. However, too much significance should not be attached to the latter since: (a) the comparison is based on an extrapolation of 100 to 300 K and (b) the constant heat capacity reported by Coughlin and King [7] is not realistic.

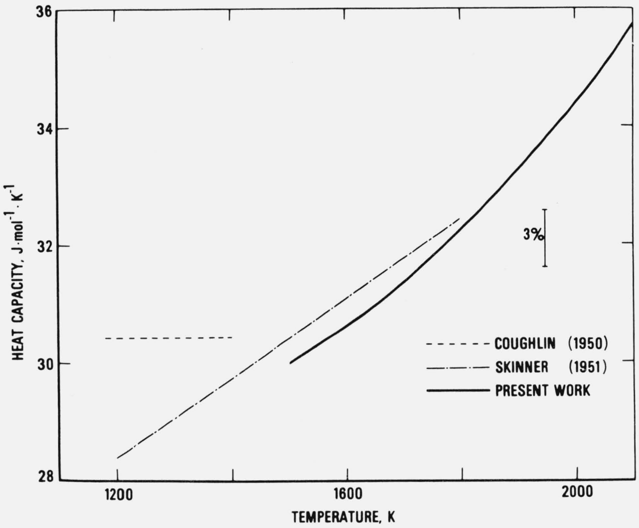

FIGURE 1. *Heat capacity of zirconium reported in the literature.*

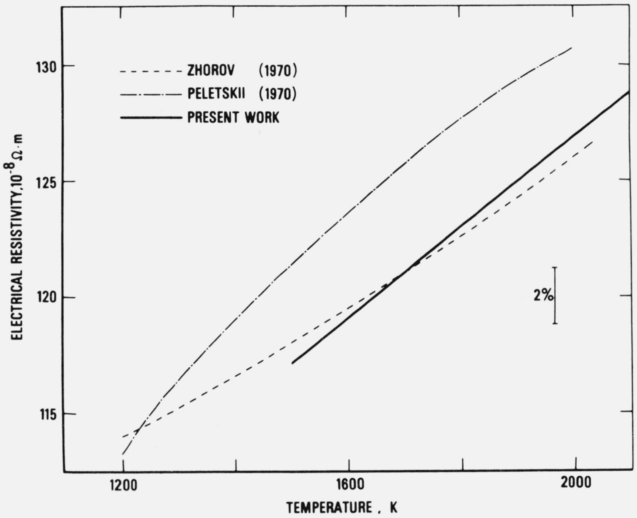

FIGURE 2. *Electrical resistivity of zirconium reported in the literature.*

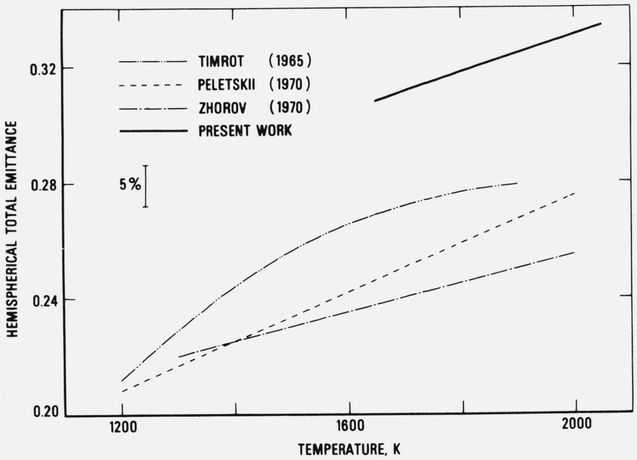

FIGURE 3. *Hemispherical total emittance of zirconium reported in the literature.*

The electrical resistivity results are in reasonably good agreement (maximum difference less than 1 percent) with those of Zhorov [8], and are approximately 3–4 percent lower than those of Peletskii et al. [9], in the overlapping temperature regions.

Zirconium undergoes a solid-solid phase transformation around 1150 K. The measurements of the geometrical quantities of the specimen after a number of experiments indicated permanent distortions (elongation) due to repeated heating and cooling through the transformation point. The reported electrical resistivity results are corrected for the permanent geometrical changes. The magnitude of this correction was about 1 percent. At 293 K, the average electrical resistivity (42.8×10^{-8} Ω · m) of the three specimens used in this work is within 3.5 percent of the values reported in the literature by Adenstedt [10] (44.1×10^{-8} Ω · m) and by Powell and Tye [11] (42.2×10^{-8} Ω · m and 44.3×10^{-8} Ω · m). Some of the differences in the electrical resistivity values may be due to differences in the chemical composition of the specimens.

The hemispherical total emittance values reported in this work are higher (10–25%) than those reported by Timrot and Peletskii [12], Peletskii et al. [9], and Zhorov [8]. Considerable differences in hemispherical total emittance results of various investigators may be due to differences in specimen surface conditions. Changes in the specimens' surface conditions were noticed during this work, with the initial smooth polished surface changing to an uneven rough surface as the result of repeated heating and cooling through the transformation point. This may partially account for the high emittance values.

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4. Appendix

TABLE A–1. *Experimental results on heat capacity of zirconium*

Range	Temperature (K)	Specimen-1		Specimen-2		Specimen-3			
		C_p (J · mol^{-1} · K^{-1})	ΔC_p* (%)	C_p (J · mol^{-1} · K^{-1})	ΔC_p* (%)	C_p (J · mol^{-1} · K^{-1})	ΔC_p* (%)	C_p (J · mol^{-1} · K^{-1})	ΔC_p* (%)
I	1500	29.87	−0.52	29.72	−1.03	29.73	−1.00	30.02	−0.02
	1550	30.38	+0.20	30.16	−0.53	30.22	−0.33	30.38	+0.20
	1600	30.91	+0.86	30.61	−0.11	30.72	+0.25	30.72	+0.25
	1650	31.45	+1.42	31.05	+0.15	31.22	+0.69	31.65	+2.04
II	1700	31.54	+0.46	31.10	−0.95	31.32	−0.24	31.32	−0.24
	1750	32.05	+0.72	31.79	−0.10	31.79	−0.10	31.83	+0.03
	1800	32.57	+0.90	32.22	−0.18	32.25	−0.09	32.36	+0.25
	1850					32.71	−0.18	32.89	+0.37
	1900					33.17	−0.37	33.42	+0.38
III	1850	32.66	−0.34	32.42	−1.08				
	1900	33.31	+0.05	33.05	−0.74				
	1950	33.99	+0.41	33.72	−0.39				
	2000	34.70	+0.74	34.40	−0.12				
	2050	35.44	+1.05						
IV	1850	32.96	+0.58	32.77	0.00				
	1900	33.21	−0.25	33.02	−0.83				
	1950	33.63	−0.66	33.43	−1.26				
	2000	34.25	−0.56	34.01	−1.27				
	2050	35.12	+0.15	34.79	−0.79				
	2100	36.31	+1.62	35.82	+0.27				

*The quantity ΔC_p is percentage deviation of the individual results from the smooth function represented by the pertinent equation in table 3.

TABLE A–2. *Experimental results on electrical resistivity of zirconium*

Range	Temperature (K)	Specimen-1		Specimen-2		Specimen-3			
		ρ (10^{-8} Ω · m)	$\Delta\rho$* (%)	ρ (10^{-8} Ω · m)	$\Delta\rho$* (%)	ρ (10^{-8} Ω · m)	$\Delta\rho$* (%)	ρ (10^{-8} Ω · m)	$\Delta\rho$* (%)
I	1500	115.97	−1.02	116.07	−0.93	117.55	+0.34	117.67	+0.44
	1550	117.06	−0.91	117.16	−0.82	118.62	+0.42	118.85	+0.61
	1600	118.14	−0.81	118.30	−0.67	119.75	+0.54	119.98	+0.74
	1650	119.25	−0.69	119.39	−0.57	120.87	+0.66	121.10	+0.85
II	1700	120.36	−0.57	120.50	−0.45	122.00	+0.78	122.00	+0.78
	1750	121.41	−0.50	121.56	−0.38	123.06	+0.85	123.07	+0.86
	1800	122.50	−0.40	122.57	−0.34	124.10	+0.89	124.10	+0.89
	1850					125.13	+0.93	125.13	+0.93
	1900					126.14	+0.95	126.23	+1.02
III	1850	123.60	−0.29	123.61	−0.29				
	1900	124.62	−0.25	124.60	−0.27				
	1950	125.65	−0.21	125.59	−0.26				
	2000	126.68	−0.16	126.58	−0.24				
	2050	127.71	−0.12						
IV	1850	123.60	−0.29	123.61	−0.29				
	1900	124.64	−0.24	124.67	−0.21				
	1950	125.65	−0.21	125.68	−0.18				
	2000	126.63	−0.20	126.66	−0.18				
	2050	127.61	−0.19	127.62	−0.19				
	2100	128.61	−0.17	128.59	−0.19				

*The quantity $\Delta\rho$ is percentage deviation of the individual results from the smooth function represented by the pertinent equation in table 3.

TABLE A–3. *Experimental results on hemispherical total emittance of zirconium*

Specimen–1			Specimen–2		
Temperature (K)	ϵ	$\Delta\epsilon$ [a] (%)	Temperature (K)	ϵ	$\Delta\epsilon$ [a] (%)
1674.7	0.307	−0.87	1650.5	0.301	−2.37
1680.1	.309	−0.33	1655.5	.304	−1.47
1685.7	.310	−0.12	1660.7	.307	−0.58
1691.4	.312	+0.40	1666.0	.310	+0.28
1697.1	.313	+0.61	1671.4	.312	+0.81
1703.0	.314	+0.80	1676.9	.315	+1.65
1848.8	.320	−0.24	1980.7	.326	−0.96
1856.9	.322	+0.23	1991.0	.327	−0.86
1865.3	.324	+0.68	2001.6	.328	−0.75
1873.7	.325	+0.82	2012.3	.329	−0.65
1882.4	.327	+1.26	2023.3	.329	−0.87
1891.3	.328	+1.39	2034.5	.330	−0.78
2003.3	.330	−0.18			
2014.3	.332	+0.22			
2025.6	.333	+0.30			
2037.1	.334	+0.38			
2048.9	.335	+0.45			
2061.0	.336	+0.52			

[a] The quantity $\Delta\epsilon$ is percentage deviation of the individual results from the smooth function represented by the pertinent equation in table 3.

5. References

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