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High-Speed (Subsecond) Measurement of Heat Capacity, Electrical Resistivity, and Thermal Radiation Properties of Tungsten in the Range 2000 to 3600 K*

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Measurements of heat capacity, electrical resistivity, hemispherical total emittance, and normal spectral emittance of tungsten above 2000 K by a pulse heating technique are described. Duration of an individual experiment, in which the specimen is heated from room temperature to near its melting point, is less than one second. Temperature measurements are made with a photoelectric pyrometer. Experimental quantities are recorded with a digital data acquisition system, which has a full-scale signal resolution of one part in 8000. Time resolution of the entire system is 0.4 ms. Results on the above properties of tungsten in the range 2000 to 3600 K are reported and are compared with those in the literature. Estimated inaccuracy of measured properties in the above temperature range is: 2 to 3 percent for heat capacity, 1 percent for electrical resistivity, 3 percent for hemispherical total and normal spectral emittances.

Key words: Electrical resistivity; emittance; heat capacity; high-speed measurements; high temperature; thermal radiation properties; thermodynamics; tungsten.

1. Introduction

Tungsten has the highest melting point (above 3600 K) of any known metal. Because of the difficulties involved in performing accurate experiments by conventional techniques at temperatures above approximately 2500 K, a high-speed method was developed to measure heat capacity, electrical resistivity, hemispherical total emittance and normal spectral emittance of electrical conductors. In this paper, application of this technique to measurements on tungsten in the temperature range 2000 to 3600 K is described.

The method is based on rapid resistive self-heating of the specimen from room temperature to near its melting point. During the short experiment, which lasts less than 1 s, current flowing through the specimen, potential across the specimen and specimen temperature are measured. Temperature measurements are made with a high-speed photoelectric pyrometer [1].[1] Recordings of experimental quantities are made with a digital data acquisition system, which has a time resolution of 0.4 ms, and a full-scale signal resolution of one part in 8000. Details regarding the construction and operation of the measurement system, and other pertinent information, such as formulation of relations for properties etc., are given in earlier publications [2, 3] in connection with measurements on molybdenum and tantalum.

2. Measurements

The measurements were made in the temperature interval 1900 to 3600 K. To optimize the operation of the pyrometer, this temperature interval was divided into four ranges: low, 1900 to 2200 K; medium, 2150 to 2500 K; high, 2450 to 2900 K; and very high, 2850 to 3600 K. Two experiments were conducted in each range; and three additional experiments were conducted in the first three ranges in which the surface radiance of the specimen was measured. Before the start of the experiments, the specimen was annealed by subjecting it to approximately 30 heating pulses (up to 3200 K).

The duration of the current pulses in the experiments ranged from 410 to 630 ms depending on the desired final temperature. The average heating rate of the specimen was: 7100 K s^{-1} at 2000 K, 5600 K s^{-1} at 3000 K, and 3700 K s^{-1} at 3600 K. At these temperatures, radiative heat losses from the specimen amounted to approximately 3, 12, and 27 percent of the input power, respectively. All of the experiments were conducted with the specimen in a vacuum environment of approximately 10^{-4} torr.

The data on voltage, current, and temperature were used to obtain third degree polynomial functions for

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

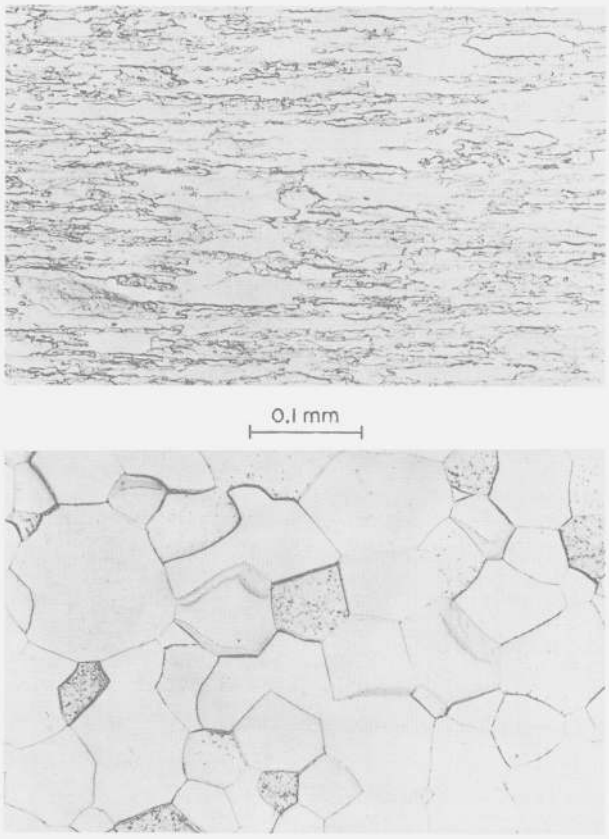

FIGURE 1. *Photomicrographs of the tungsten specimen before (upper photograph) and after (lower photograph) the entire set of experiments.*

each quantity in terms of time, which then provided the input information for the determination of properties.

The pyrometer was calibrated before and after the entire set of experiments against a tungsten-filament standard lamp, which in turn was calibrated against the NBS temperature standard. The digital recording system, including the differential amplifiers, was also calibrated before and after the entire set of experiments. The details of the calibration procedures are given in an earlier publication [2].

The specimen was a tube fabricated from a tungsten rod by removing the center portion by an electro-erosion technique. The outer surface of the specimen was polished to reduce heat loss due to thermal radiation. The nominal dimensions of the specimen were: length, 4 in (101 mm); outside diameter, 0.25 in (6.3 mm); and wall thickness, 0.02 in (0.5 mm).

Specimen characterization was made by the following methods: photomicrography, spectrochemical analysis, and residual resistivity ratio. Photomicrographs of the specimen (figure 1) indicate that considerable grain growth took place as the result of pulse heating to high temperatures. A list of the nature and composition of impurities in the specimen, at the end of the entire set of experiments as determined by spectrochemical analysis,[2] is given in table 1. The residual resistivity ratio of the specimen (ratio of electrical resistivity at 273 K to that at 4 K), measured before the experiments, was 41.

TABLE 1. *Impurities in tungsten specimen*

Impurity	Composition, ppm (by weight)
Al	5
B	< 2
Ca	15
Cr	5
Co	< 2
Cu	10
Fe	60
Mg	< 2
Mn	< 2
Mo	310
Nb	< 20
Ni	< 2
Pb	< 2
Si	5
Sn	< 2
Sr	< 2
Th	< 250
Ti	10
Zr	30

$450 < \text{Total} < 740$

The "effective" mass of the specimen was calculated from the total mass by the ratio of the geometric surface area between voltage probes to total surface area. Length measurements at room temperature were made with a micrometer microscope. The cross-sectional area of the specimen was calculated from the mass, density, and geometry. Density of the tungsten specimen was measured at 293 K to be 19.23×10^3 kg m^{-3}. This compares favorably with a previously cited value of 19.3×10^3 kg m^{-3} [4].

3. Experimental Results

This section presents the thermophysical properties determined from the measured quantities. All values are based on the 1968 International Practical Temperature Scale [5]. In all computations, the geometrical quantities are based on their room temperature (298 K) dimensions. The experimental results are represented by polynomial functions in temperature obtained by least squares approximation of the individual points. The final values on properties at 100 degree temperature intervals computed using the functions are presented in table 2. Results obtained from individual experiments, by the method described previously [2], are given in the appendix (tables A–1, A–2, and A–3). The patterns of deviations of individual data points from the smooth functions for the properties are similar to those in the earlier work on tantalum [3].

[2] Spectrochemical analysis of the tungsten specimen was made by the Lamp Metals and Components Department of the General Electric Company.

TABLE 2. *Heat capacity, electrical resistivity, hemispherical total emittance and normal spectral emittance of tungsten*

Temp. K	c_p J mol^{-1} K^{-1}	ρ [a] 10^{-8} Ω m	ϵ [a]	$\epsilon_{N,\lambda}$
2000	31.65	56.22	[b] 0.318	0.379
2100	32.49	59.74	[b] .321	.379
2200	33.29	63.25	[b] .324	.379
2300	34.08	66.77	.326	.379
2400	34.89	70.28	.329	.379
2500	35.72	73.80	.332	.379
2600	36.61	77.31	.335	.379
2700	37.57	80.83	.338	.379
2800	38.63	84.34	.340	.379
2900	39.81	87.86	.343	.379
3000	41.14	91.37	.346	.379
3100	42.62	94.89	.349	
3200	44.29	98.40	.351	
3300	46.17	101.92	.354	
3400	48.27	105.43	.357	
3500	50.63	108.95		
3600	53.25	112.46		

[a] Based on ambient-temperature (298 K) dimensions.
[b] Extrapolated from higher temperature results.

3.1. Heat Capacity

Heat capacity was computed from data taken during the heating period. A correction for power loss due to thermal radiation was made using the results on hemispherical total emittance. The function for heat capacity (standard deviation = 0.7%) that represents the results in the temperature range 2000 to 3600 K is:

$$c_p = -25.71 + 6.331 \times 10^{-2} T - 2.459 \times 10^{-5} T^2 + 3.638 \times 10^{-9} T^3 \quad (1)$$

where T is in K and c_p in J mol^{-1}K^{-1}. In the computations of the heat capacity, the atomic weight of tungsten was taken as 183.85.

To determine the effect of thermal cycling on heat capacity, the results of four additional experiments covering the range 2000 to 3300 K were compared with those reported above. The average absolute difference between the two sets of results was less than 0.1 percent, which is smaller than the measurement resolution. This indicates that the measurements were not sensitive to thermal cycling.

3.2. Electrical Resistivity

The electrical resistivity of tungsten was determined from the same experiments that were used to calculate the heat capacity. The function for electrical resistivity (standard deviation = 0.4%) that represents the results in the temperature range 2000 to 3600 K is:

$$\rho = -14.08 + 3.515 \times 10^{-2} T \quad (2)$$

where T is in K and ρ in 10^{-8} Ωm. The results of thermal cycling indicate an average absolute difference of less than 0.5 percent in electrical resistivity. The measurement, before the pulse experiments, of the electrical resistivity of the specimen at 293 K with a Kelvin bridge yielded a value of 5.45×10^{-8} Ωm.

3.3. Hemispherical Total Emittance

Hemispherical total emittance was computed using data taken during both heating and initial free cooling periods. The function for hemispherical total emittance (standard deviation = 1%) that represents the results in the temperature range 2300 to 3400 K is:

$$\epsilon = 0.2627 + 2.770 \times 10^{-5} T \quad (3)$$

where T is in K.

3.4. Normal Spectral Emittance

Normal spectral emittance was computed using data from three sets of two experiments, one in which the pyrometer was aimed at the surface of the specimen, and another in which it was aimed at the blackbody hole in the specimen. The target on the surface was a narrow flat surface ground along the specimen. The measurements were made at the effective wavelength of the pyrometer interference filter (650 nm; bandwidth 10 nm). The function for normal spectral emittance (standard deviation = 0.2%) that represents the results in the temperature range 2000 to 3000 K is:

$$\epsilon_{N,\lambda} = 0.3804 - 5.060 \times 10^{-7} T \quad (4)$$

where T is in K.

4. Estimate of Errors

Estimates of errors in measured and computed quantities lead to the following estimates of errors in the properties over the temperature range 2000 to 3600 K.

Heat capacity: 2 percent at 2000 K, 3 percent at 3600 K.
Electrical resistivity: 1 percent
Hemispherical total emittance: 3 percent
Normal spectral emittance: 3 percent

Details regarding the estimates of errors and their combination in high-speed experiments using the present measurement system are given in a previous publication [2]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication.

5. Discussion

The heat capacity and electrical resistivity results of this work are compared graphically with those in the literature in figures 2 and 3, respectively. Numerical comparisons are given in tables 3 and 4. It may be

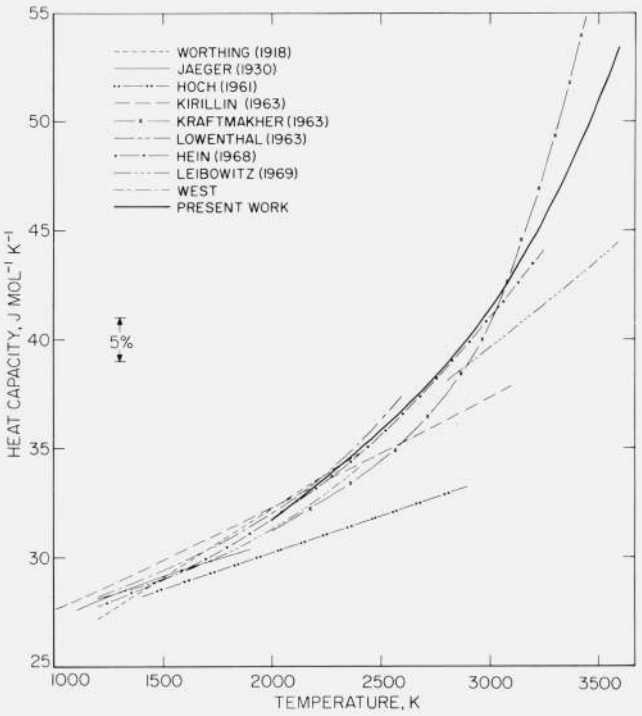

FIGURE 2. *Heat capacity of tungsten reported in the literature.*

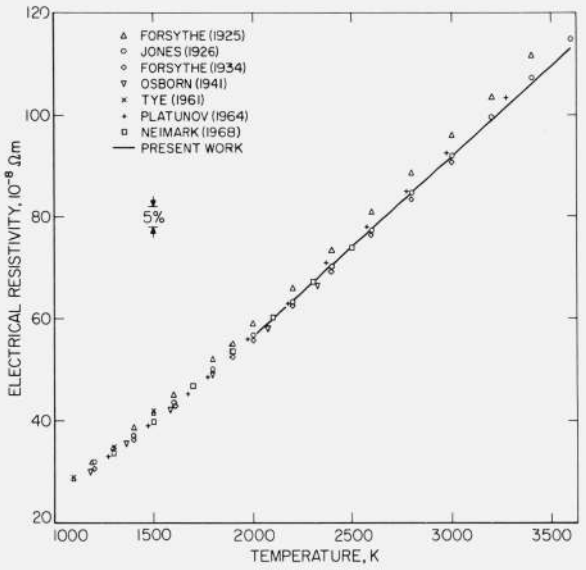

FIGURE 3. *Electrical resistivity of tungsten reported in the literature.*

TABLE 3. *Tungsten heat capacity difference (previous literature values minus present work values) in percent*

Investigator	Ref.	Year	Method	Temperature, K								
				2000	2200	2400	2600	2800	3000	3200	3400	3600
Worthing	10	1918	pulse	+1.6	+0.3	−0.7						
Jaeger and Rosenbohm	11	1930	drop	[a]−3.2								
Hoch and Johnston	12	1961	drop	−4.7	−7.3	−9.7	−12	−15				
Kirillin et al	13	1963	drop	+1.6	−0.4	−2.1	−4.0	−6.7	−9.7			
Kraftmakher and Strelkov	14	1963	modul.	−1.4	−2.4	−3.6	−4.0	−3.2	−0.8	+3.4	+9.5	+17
Lowenthal	15	1963	modul.	−1.5	−2.3	−2.4						
Hein and Flagella	16	1968	drop	−.03	−0.7	−0.6	−0.2	−0.1	−0.7	−2.4		
Leibowitz et al	17	1968	drop					−1.8	−4.1	−7.5	−12	−17
West and Ishihara	18		drop	+1.0	+0.4	+0.8	+1.9					

[a] Extrapolated from 1873 K.

seen that most of the results are in general agreement at 2000 K. Considerable disagreement in heat capacity exists above 2500 K. This may be expected, since above this temperature accuracy of heat capacity measured by conventional methods decreases rapidly. Estimates of errors in papers cited lead to an estimate of inaccuracies in previously reported heat capacity and electrical resistivity of approximately 5 to 15 and 1 to 5 percent, respectively, in the temperature range considered. The present result of the electrical resistivity of tungsten corresponding to 293 K, as well as values reported in the literature, are given in table 5.

TABLE 4. *Tungsten electrical resistivity difference (previous literature values minus present work) in percent*

Investigator	Ref.	Year	Temperature, K								
			2000	2200	2400	2600	2800	3000	3200	3400	3600
Forsythe and Worthing	19	1925	+5.1	+4.7	+4.6	+4.8	+4.9	+5.2	+5.5	+5.9	
Jones	20	1926	+0.8	+0.2	+0.1	+0.1	+0.4	+0.7	+1.2	+1.7	+2.2
Forsythe and Watson	32	1934	−0.9	−1.3	−1.5	−1.4	−1.3	−1.1			
Osborn	21	1941	−0.4	−0.6							
Platunov and Fedorov	22	1964	+1.1	+1.2	+1.9	+2.2	+2.4	+2.5	+2.5		
Neimark and Voronin	23	1967	+1.2	+0.7	+0.5						

TABLE 5. *Electrical resistivity of tungsten at 293 K*

Investigator	Ref.	Year	Resistivity 10^{-8} Ω m
Forsythe and Worthing	19	1925	5.46
Jones	20	1926	5.49
Forsythe and Watson	32	1934	5.50
White and Woods	31	1959	[a] 5.29
Tye	24	1961	5.45
Present work			5.45

[a] Ideal resistivity.

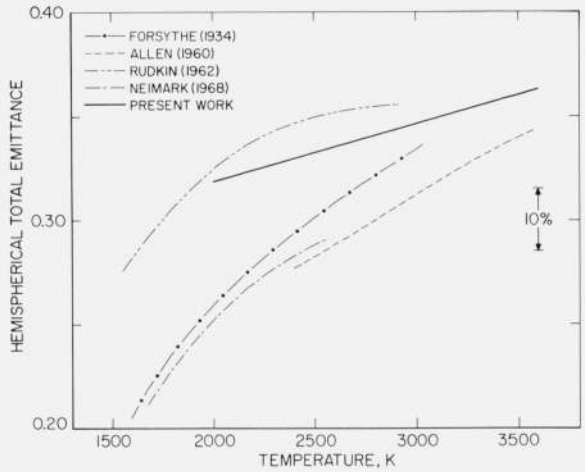

FIGURE 4. *Hemispherical total emittance of tungsten reported in the literature.*

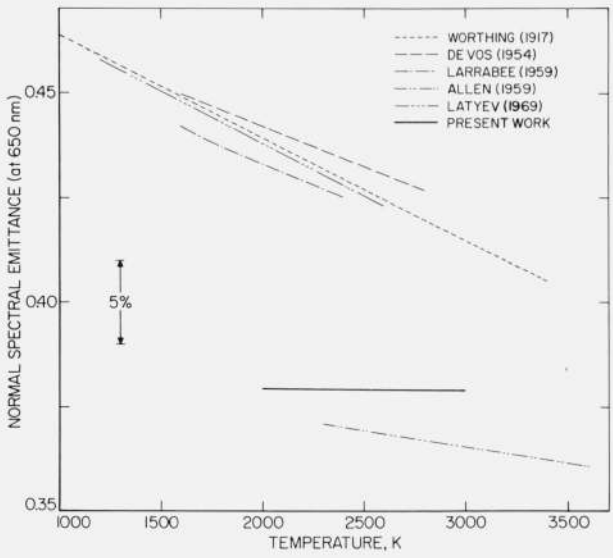

FIGURE 5. *Normal spectral emittance of tungsten at λ=650 nm reported in the literature.*

The results for hemispherical total emittance and normal spectral emittance of this work and those in the literature are presented in figures 4 and 5, respectively. Because of the strong dependence of emittance on surface conditions, considerable deviations exist in the results of various investigators.

Heat capacity results at high temperatures are considerably higher than the Dulong and Petit value of $3R$. Some of this departure is due to $c_p - c_v$ and the electronic terms. However, they do not account for the entire departure. Heat capacity above the Debye temperature may be expressed by

$$c_p = A - \frac{B}{T^2} + CT + \Delta c \qquad (5)$$

where the constant term is $3R$ (24.943 J mol^{-1}K^{-1}), the term in T^{-2} is the first term in the expansion of the Debye function, the term in T represents $c_p - c_v$ and electronic contributions, and the quantity Δc represents excess in measured heat capacity at high temperatures, which is not accounted for by the first three terms. The coefficients $B(7.72 \times 10^4)$ and $C(2.33 \times 10^{-3})$ were obtained from data on heat capacity at room and moderate temperatures (at 298.15 and 1000 K) given by Hultgren et al. [6].

TABLE 6. *Excess heat capacity* Δc *in eq (5) and estimated vacancy contribution to heat capacity of tungsten*

T	Δc	c_{vac}
K	*J mol*$^{-1}$ *K*$^{-1}$	*J mol*$^{-1}$ *K*$^{-1}$
2000	2.07	0.0005
2200	3.24	.002
2400	4.37	.009
2600	5.62	.03
2800	7.18	.06
3000	9.21	.14
3200	11.90	.26
3400	15.41	.47
3600	19.93	.79

Using eq (5) and the heat capacity results of this work, the quantity Δc was computed for temperatures above 2000 K. The results are tabulated in table 6. The uncertainty in the computed Δc may be as high as 1 J mol^{-1} K^{-1}. This was obtained from the combined uncertainties in the coefficients in eq (5) and the measured heat capacities.

Although the mechanisms of vacancy generation become important at high temperatures, it was not possible to attribute the high values entirely to vacancies. To demonstrate this, a crude estimate of the contribution of vacancies to heat capacity was made using the method described in a previous publication [2]. The reported values for vacancy formation energy of tungsten are 3.3 eV [7] and 3.6 eV [8]. Results of quenching experiments on various refractory elements [7, 9] have indicated that vacancy concentrations are probably in the range 0.01 to 0.1 percent at their melting points. Estimates corresponding to a vacancy concentration of 0.1 percent at the melting point and a vacancy formation energy of 3.3 eV are given in table 6. The results indicate that vacancy contribution would be small, less than 0.8 J mol^{-1} K^{-1} (upper limit) at 3600 K, and would not account for the high heat capacity values.

If the entire difference between measured and computed [using the first three terms in eq (5)] heat capacities is attributed to vacancies, values of 1.3 eV for energy and 12 percent for concentration at the melting point are obtained. Both of these values seem to be unrealistic for tungsten.

To give a simple expression for the heat capacity of tungsten over a wide temperature range, an empirical

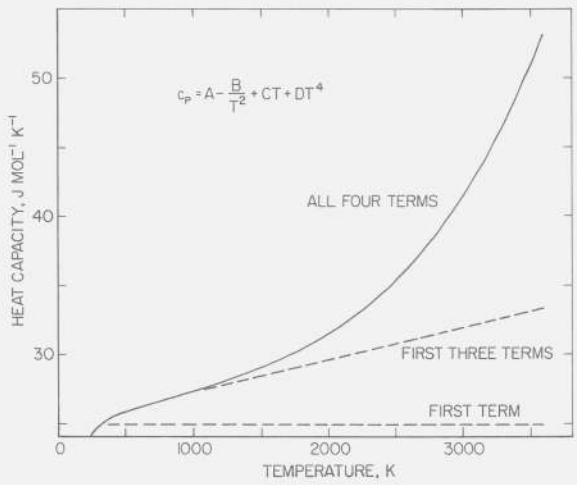

FIGURE 6. *Heat capacity of tungsten according to eq (6).*

term in T^4 for the quantity Δc in eq (5) was substituted. The coefficient of this term was obtained from the results of the present work in conjunction with the values given by Hultgren et al. [6] at temperatures below 1000 K. Then, eq (5) for the range 300 to 3600 K becomes

$$c_p = 24.943 - \frac{7.72 \times 10^4}{T^2} + 2.33 \times 10^{-3} T + 1.18 \times 10^{-13} T^4 \qquad (6)$$

where T is in K and c_p in J mol^{-1} K^{-1}. Average absolute deviation of the individual points from the function over the temperature range considered is 0.2 percent. Equation (6) is presented graphically in figure 6.

The experimental results reported in this paper have further substantiated the feasibility of accurate simultaneous measurement of selected properties above 2000 K by a millisecond resolution pulse method.

The authors express their gratitude to C. W. Beckett for his interest and encouragement of research in high-speed methods of measuring thermophysical properties. They also extend their appreciation to M. S. Morse for his contribution in connection with electronic instrumentation, which is a vital part of the entire measurement system.

6. Appendix

TABLE A–1. *Experimental results on heat capacity and electrical resistivity of tungsten* [a]

Range	Run	1		2	
	T	c_p	ρ	c_p	ρ
	1900	31.37	53.01	30.84	53.03
	1950	31.48	54.65	31.28	54.66
	2000	31.68	56.30	31.69	56.30
Low	2050	31.98	57.96	32.09	57.96
	2100	32.39	59.63	32.48	59.63
	2150	32.90	61.31	32.84	61.32
	2200	33.54	63.03	33.18	63.03
	2150	32.68	61.40	32.24	61.43
	2200	33.06	63.10	32.80	63.10
	2250	33.47	64.82	33.36	64.80
Medium	2300	33.91	66.54	33.92	66.52
	2350	34.38	68.28	34.47	68.28
	2400	34.90	70.04	35.03	70.05
	2450	35.47	71.82	35.58	71.85
	2500	36.10	73.62	36.12	73.66
	2450	34.93	71.79	35.03	71.79
	2500	35.46	73.57	35.55	73.58
	2550	36.00	75.36	36.07	75.39
	2600	36.55	77.17	36.62	77.20
High	2650	37.13	78.98	37.19	79.02
	2700	37.73	80.80	37.78	80.85
	2750	38.36	82.62	38.40	82.68
	2800	39.03	84.45	39.05	84.51
	2850	39.73	86.27	39.75	86.33
	2900	40.48	88.09	40.49	88.15
	2850	39.00	86.61	38.94	86.72
	2900	39.64	88.38	39.62	88.48
	2950	40.31	90.14	40.31	90.24
	3000	41.00	91.88	41.02	91.98
	3050	41.72	93.61	41.76	93.70
	3100	42.47	95.32	42.52	95.40
	3150	43.25	97.00	43.32	97.09
Very high	3200	44.08	98.66	44.15	98.75
	3250	44.96	100.30	45.03	100.39
	3300	45.89	101.91	45.98	102.00
	3350	46.89	103.50	46.99	103.59
	3400	47.97	105.06	48.09	105.14
	3450			49.31	106.66
	3500			50.67	108.14
	3550			52.21	109.59
	3600			54.00	111.01

[a] Temperature in K; heat capacity in J mol^{-1} K^{-1}; electrical resistivity in 10^{-8} Ω m.

TABLE A–2. *Experimental results on hemispherical total emittance of tungsten*

T K	ϵ
2333	0.323
2336	.327
2336	.325
2339	.328
2668	.336
2673	.340
2673	.339
2678	.343
3005	.342
3012	.346
3013	.346
3020	.349
3312	.347
3323	.352
3323	.351
3334	.356
3407	.360
3418	.355
3418	.362
3430	.360

TABLE A–3. *Experimental results on normal spectral emittance of tungsten at* $\lambda = 650$ *nm*

T K	$\epsilon_{N,\lambda}$
2076	0.380
2111	.379
2146	.379
2180	.379
2214	.379
2248	.381
2339	.378
2393	.378
2447	.379
2499	.379
2551	.380
2600	.380
2670	.379
2739	.378
2805	.379
2870	.379
2933	.380

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