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Thermal Properties of Tantalum Between 300 and 2300 K[1]

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A subsecond pulse heating method was applied to measure the specific heat capacity, electrical resistivity, total hemispherical emissivity, and normal spectral emissivity of 99.9% pure tantalum in the form of 2-mm-diameter wire. W/Re thermocouple thermometry was applied from 300 to 2300 K, with emissivity measurements above 1300 K involving pyrometric measurements. The maximum uncertainties in the specific heat capacity and electrical resistivity were less than 3 and 1% respectively. The uncertainty of emissivity measurements was estimated as ±5%. The results are compared with literature values.

KEY WORDS: electrical conductors; electrical resistivity; hemispherical total emissivity; high temperatures; normal spectral emissivity; refractory metals; specific heat; tantalum.

1. INTRODUCTION

Measurements of tantalum represent part of a comprehensive study of thermal properties of refractory metals at the Institute of Nuclear Sciences Vinča. Calorimetric, electrical, and thermal optical properties of W, Mo, Nb, and Va have been studied employing millisecond pulse calorimetry [1–4], and thermal transport properties of W and Mo were studied using a laser-pulse thermal diffusivity technique [5, 6]. Current measurements of specific heat capacity, electrical resistivity, total hemispherical emissivity, and normal spectral emissivity of tantalum are reported.

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2. METHOD

The millisecond pulse calorimetry method with direct heating using thermocouple thermometry is based on rapid heating of a sample in the form of a thin rod to the desired temperature. During the heating of the sample, the data on current and voltage drop over the effective measurement zone of the sample are collected, together with the EMF of the thermocouple welded intrinsically at the center of the effective measurement zone. The thermocouple EMF is also recorded during the initial portion of the sample cooling. The method is described in detail in Refs. 7 and 8.

3. EXPERIMENTAL

3.1. Sample

The sample of tantalum was supplied by Goodfellow, UK, with a nominal impurity content (ppm) as follows: Al, 5; Ca, 2; Co, 1; Cr, 5; Cu, 2; Fe, 30; Mg, 5; Mn, 2; Mo, 100; Na, 10; Nb, <500; Ni, 3; Si, 10; Sn, 2; Ti, 20; V, 5; W, 100; and Zr, 10 (as stated by the manufacturer). It was used in the form of a rod 1.90 mm in diameter and either 200 or 110 mm in length. The sample was used in measurements without additional thermal treatment.

3.2. Measurements

Temperature measurements for the specific heat and electrical resistivity determinations were carried out with 5% WRe/26% WRe thermocouples, with wires 100 or 75 μm in diameter. DC current pulses were delivered by two heavy-duty 12-V batteries connected in series, resulting in heating rates from 1000 to 2800 $K \cdot s^{-1}$ (depending on the series resistances). The current through the sample was measured using a 1-mΩ standard resistor.

Temperature data during heating and cooling periods, which were obtained with parallel thermocouple and pyrometer measurements, enabled determinations of the hemispherical total emissivity and the normal spectral emissivity. The experimental procedure is described in detail in Refs. 7 and 8.

4. RESULTS

4.1. Summary

A total of 40 specific heat and electrical resistivity measurements was made, with current ranging between 450 and 900 A. At the lower heating rates, the presence of axial heat losses from the effective sample length was registered. The heating rates were increased until the temperature remained uniform over the effective sample length during the whole experiment. This limited the length of experiments to 1000 ms. Experiments covered the range between room temperature and about 2300 K.

Each individual measurement resulted in a continuous set of specific heat and electrical resistivity data, covering the whole, or a part of the temperature range. Of 40 measurements, about 20 were adequate for further processing. All the raw data, specific heat and electrical resistivity, were used to obtain a single least-squares fitted polynomial for both properties.

The specific heat is represented by a polynomial function of the third degree,

$$C_p = 134.64 + 2.7981 \times 10^{-2} T - 1.5971 \times 10^{-5} T^2 + 5.8259 \times 10^{-9} T^3 \quad (1)$$

All individual specific heat measurements lie $\pm 3\%$ from Eq. (1) over most of the range, reaching about $\pm 5\%$ at 2300 K.

The polynomial representing electrical resistivity has the form

$$\begin{aligned} \rho = & -1.03 \times 10^{-8} + 5.1923 \times 10^{-10} T \\ & - 6.3911 \times 10^{-14} T^2 + 5.1236 \times 10^{-18} T^3 \end{aligned} \quad (2)$$

with individual electrical resistivity measurements deviating from the fitting function by $\pm 3\%$ at 300 K and by about $\pm 2\%$ over the rest of the range.

Before the first and after the last experiment, room-temperature electrical resistivities were measured by the stationary-state four-probe-with-current-reversal method. Table I contains values of both functions in 100 K increments, the measured room-temperature electrical resistivities and the electrical resistivity value stated by the sample manufacturer.

Data collected during pulse experiments in the high-temperature range enabled determination of both hemispherical total and normal spectral emissivities [7, 8]. The data were collected simultaneously by an intristic thermocouple and an optical pyrometer, located at the center of the effective sample zone. Due to properties of the pyrometer operating at $\lambda \approx 900$ nm, emissivity could be measured only above about 1000 K. The averaged hemispherical total emissivity follows a linear function, and it is represented by

$$\varepsilon_t = -0.06835 + 1.8524 \times 10^{-4} T \quad (3)$$

Table I. Experimental Results on Thermophysical Properties of Tantalum[a]

Temperature (K)	Specific Heat ($J \cdot kg^{-1} \cdot K^{-1}$)	Electrical resistivity ($10^{-7}\,\Omega \cdot m$)	Hemispherical total emissivity	Spectral normal emissivity
293.15	—	1.350[b]	—	—
291.95	—	1.298[c]	—	—
298.65	—	1.403[d]	—	—
300	141.8	1.399	—	—
400	143.6	1.875	—	—
500	145.4	2.340	—	—
600	146.9	2.793	—	—
700	148.4	3.236	—	—
800	149.8	3.668	—	—
900	151.1	4.090	—	—
1000	152.5	4.501	—	—
1100	153.8	4.903	0.1354	0.33001
1200	155.3	5.296	0.1539	0.32867
1300	156.8	5.679	0.1725	0.32745
1400	158.5	6.054	0.1910	0.32635
1500	160.3	6.420	0.2095	0.32537
1600	162.4	6.778	0.2280	0.32450
1700	164.7	7.129	0.2466	0.32375
1800	167.2	7.471	0.2651	0.32311
1900	170.1	7.807	0.2836	0.32259
2000	173.3	8.135	0.3021	0.32219
2100	176.9	8.457	0.3207	0.32190
2200	180.9	8.772	0.3392	0.32173
2300	185.4	9.082	0.3577	0.32168

[a] Values from fitted polynomials.

[b] Room-temperature value as provided by the sample manufacturer.

[c] Room-temperature value obtained using the four-probe method with current reversal, measured before the first experiment.

[d] Room-temperature value obtained using the four-probe method with current reversal, measured after the final experiment.

The spectral normal emissivity decreases with temperature, following a second-order function,

$$\varepsilon_n = 0.35236 - 2.6721 \times 10^{-5} T + 5.8179 \times 10^{-9} T^2 \tag{4}$$

Numerical values of the emissivities are also presented in Table I in 100-K increments.

4.2. Uncertainties

The maximum uncertainties in determining specific heat capacity, electrical resistivity, and emissivity by the millisecond-resolution pulse technique were estimated as ±3, ±1, and ±5%, respectively [7, 8]. In this work, however, uncertainties for specific heat and electrical resistivity measurement were less. The averaging procedure, with initially small deviations of individual results from Eqs. (1) and (2), reduced the estimated maximum uncertainties.

5. DISCUSSION

5.1. Specific Heat

According to the Dulong–Petit law, the specific heat of tantalum should enter a region of saturation above the Debye temperature (which for this metal lies at about 230 K), changing in magnitude by only 30% over the next 2000 K. At elevated temperatures, however, the experimental results for tantalum exceeded such a prediction. This tendency has also been observed in our measurements on molybdenum [2], niobium [3], and vanadium [4]. Other authors investigating tantalum and other refractory metals [9–12] also observed the same phenomenon. These higher than predicted values have been attributed to electronic contributions to the specific heat [10, 12], thermal formation of lattice imperfections at high temperatures [12], and other possible causes [9].

Figure 1 compares the present results with eight sets of literature specific heat capacity data. Agreement among them depends on the temperature range. At lower temperatures the present results lie about 3% above those of Taylor and Finch [9] and of Lehman [13] and Kulish [14] and about 2% below those of Rasor and McClelland [12]. In the high-temperature range the present results virtually coincide with these of Taylor and Finch [9], lying somewhat above those of Cezairliyan et al. [11], Lowenthal [10], and Rasor and McClelland [12]. In the middle-temperature range, agreement among all data is reasonably good. The Hoch and Johnston [15] specific heat data change little with temperature, but this might be attributed to the type of polynomial used to fit their high-temperature enthalpy data. If another type, instead of a second-order polynomial was chosen, the behavior could be significantly different. The Sterrett [16] data cover only a narrow part of the middle-temperature region.

It is interesting that specific heat data discussed above have been obtained using very different measurement techniques, including three

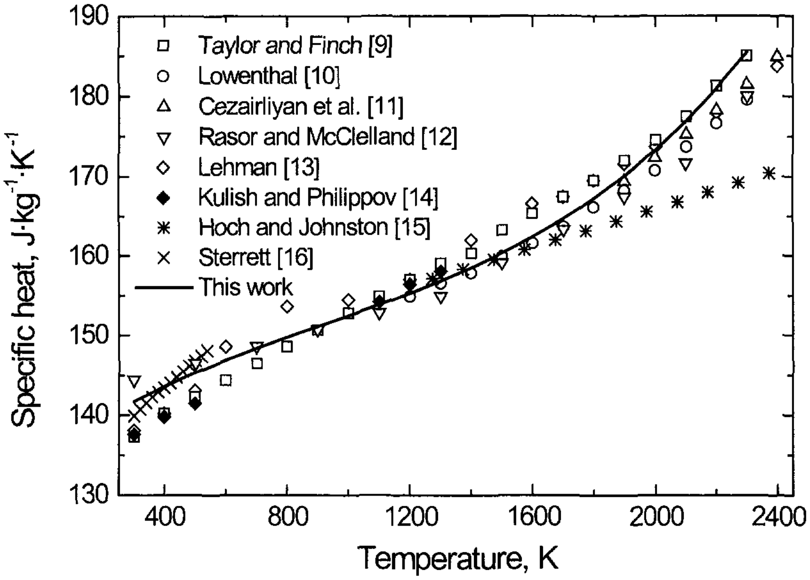

Fig. 1. Specific heat of tantalum as a function of temperature.

variations of the pulse method (using thermometry by thermocouple [7, 8], sample resistance [9, 12], and high-speed pyrometer [11]), a method of temperature oscillations [10], and a method of mixtures [16], including high-temperature vacuum drop calorimetry [15].

5.2. Electrical Resistivity

The electrical resistivity measurements are compared in Fig. 2 with the data of Tye [17], Neimark and Voronin [18], Binkele [19], and Cezairliyan et al. [11] and the present stationary-state "four-lead" room-temperature measurements. The present results are in very good agreement with other data in the lower half of the measurement range. The largest differences occurred for the data of Neimark and Voronin [18] and Cezairliyan et al. [11] at 2300 K, where their results lie about 2.5% below the present results.

5.3. Temperature Scale

Temperature measurement in this research was according to ITS-90. Different literature data in Figs. 1 and 2, however, involve values obtained with different temperature scales, IPTS-48, IPTS-68, and ITS-90. Since

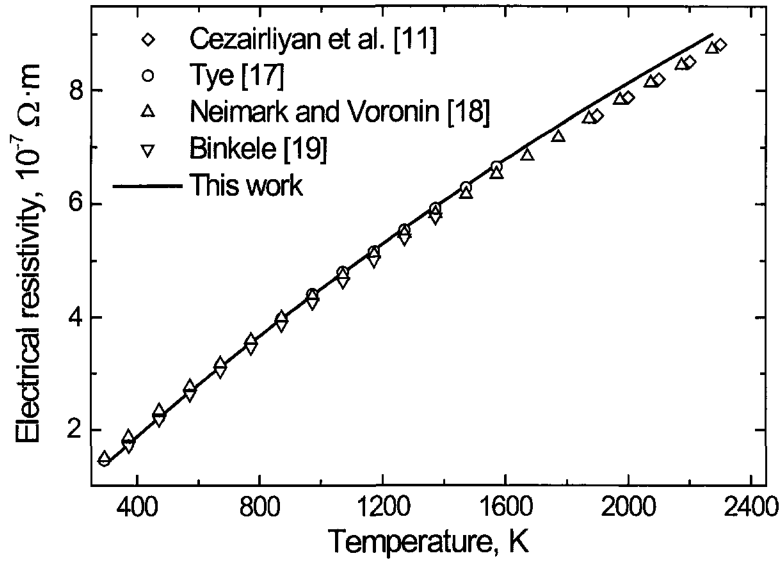

Fig. 2. Electrical resistivity of tantalum as a function of temperature.

differences among the scales are small (maximum of 4.2 K at 2300 K, IPTS-48 vs. IPTS-68), corrections are not visible on the scale of the diagrams.

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