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Electrical Resistivity of Molybdenum in the Temperature Range 1500 to 2650 K

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Measurement of the electrical resistivity of molybdenum in the temperature range 1500 to 2650 K by a subsecond-duration pulse-heating technique is described. The specimens were of the National Bureau of Standards Molybdenum Standard Reference Material 781 for Enthalpy and Heat Capacity. The electrical resistivity of molybdenum in the temperature range 1500 to 2650 K is expressed by the following function (standard deviation = 0.2%):

$$\rho = -6.7083 + 2.8949 \times 10^{-2}\, T + 5.2985 \times 10^{-7}\, T^2$$

where T is in K and ρ is in $\mu\Omega \cdot$ cm. The estimated inaccuracy in the electrical resistivity data does not exceed 1%.

KEY WORDS: dynamic measurements; electrical resistivity; high temperatures; molybdenum.

1. INTRODUCTION

Molybdenum SRM 781, certified by the National Bureau of Standards, is a standard reference material for enthalpy and heat capacity in the temperature range 273 to 2800 K [1]. The data on heat capacity above 1500 K for the molybdenum standard reference material were obtained in our laboratory with the accurate subsecond-duration, pulse-heating technique. The objective of this paper is to report the results of electrical resistivity measurements performed with the same technique, on two of the molybdenum specimens that were used for the above heat capacity measurements.

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2. METHOD

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.

The current through the specimen is determined from the measurement of the potential difference across the standard resistance placed in series with the specimen. The potential difference across the middle one-third of the specimen is measured between spring-loaded, knife-edge probes. The specimen temperature is measured at the rate of 1200 times per second with a high-speed photoelectric pyrometer [2]. The small hole in the wall at the middle of the tubular specimen provides an approximation to blackbody conditions for optical temperature measurements. The data are recorded with a digital data acquisition system every 0.4 ms with a full-scale signal 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 are given in earlier publications [3, 4].

3. MEASUREMENTS

The measurements were performed on two tubular specimens which were fabricated from adjacent portions of the same molybdenum rod.[2] An electroerosion technique was used to remove the center portion of the rod. The nominal dimensions of the specimen were: length, 76 mm; outside diameter, 6.3 mm; and wall thickness, 0.5 mm. The outer surface of the specimen was polished to reduce heat loss due to thermal radiation.

Before the start of the experiments, the specimens were heat treated by subjecting them to 20 heating pulses up to 2500 K. The temperature interval (1500 to 2650 K) was divided into five ranges in order to optimize the operation of the pyrometer. The temperature ranges (in K) were: 1500–1650, 1650–1800, 1800–2000, 2000–2250, and 2250–2650. Two series of experiments (first and second heating) were conducted on specimen 1, and one series of experiments were conducted on specimen 2, which yielded a total of

[2]The polycrystalline molybdenum rod (99.95% pure) was furnished by the Office of Standard Reference Materials (OSRM) of NBS. The details regarding the impurities are documented by OSRM.

15 experiments. All the experiments were performed with the specimen in a vacuum environment at approximately 1.3×10^{-3} N · m^{-2} ($\sim 10^{-5}$ Torr). Optical measurements performed on the chamber window before and after the pulse experiments did not show any change in its transmission. Also, weight measurements before and after the entire set of pulse experiments on a given specimen did not show any change in specimen weight.

To study the possible effects that may be attributable to the rate with which the specimen heats, seven additional experiments in the temperature range 2000 to 2250 K were performed on specimen 2 at different heating rates (1000 to 8000 K · s^{-1}). The results are discussed in the section on the estimate of errors. These experiments were performed to demonstrate the effect of heating rate on the measurements and were not used in the final computations of electrical resistivity of molybdenum.

To optimize the operation of the measurement system, the heating rate of the specimen was varied depending on the desired temperature range by adjusting the value of the resistance in series with the specimen. Duration of current pulses in the experiments (excluding the experiments that were used to check the heating-rate effect) ranged from 430 to 560 ms; and the heating rate ranged from 5400 to 6800 K · s^{-1}. The magnitude of the current pulses ranged from 1900 to 2500 A. Several times during the course of the experimental work, specimen resistance was measured at room temperature with a Kelvin bridge.

4. EXPERIMENTAL RESULTS

Electrical resistivity was calculated using the relation $\rho = RA/L$, where R (voltage/current) was the specimen resistance between the voltage probes, L was the separation between the voltage probes, and A was the specimen cross-sectional area. The cross-sectional area was determined from the measurement of specimen weight, length, and density (10.21 g · cm^{-3}).

In order to compare the electrical resistivity results of different specimens, and also of different heatings for the same specimen, data (tabulated in the Appendix) in the range 1500 to 2650 K were fitted separately for each specimen and for each heating by quadratic functions in temperature using the least squares method, giving equal weight to each point. The standard deviation of an individual point from the functions was in the range 0.14 to 0.18%. The difference among the smooth functions representing the results of experiments of different heatings for the same specimen (specimen 1) was 0.05%. The difference among the smooth functions representing the results of the measurements on specimen 2 and the averages of specimen 1 was 0.15%. Since these differences are comparable to the imprecision of the measurements, it may be concluded that no significant differences exist between the

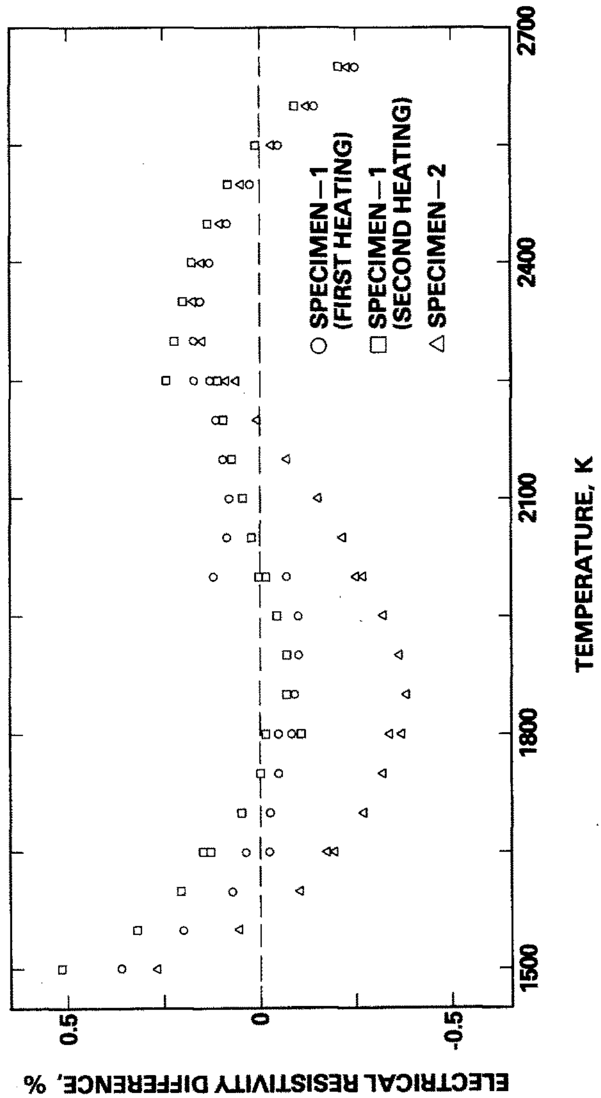

Fig. 1. Deviation of measured electrical resistivity values for molybdenum from Eq. (1).

Table I. Electrical Resistivity of Molybdenum According to Eq. (1)

T (K)	ρ ($\mu\Omega \cdot$ cm)
1,500	37.91
1,600	40.97
1,700	44.04
1,800	47.12
1,900	50.21
2,000	53.31
2,100	56.42
2,200	59.54
2,300	62.68
2,400	65.82
2,500	68.98
2,600	72.14
2,700[a]	75.32

[a]Extrapolated from 2650 K (the upper limit of the experimental data).

results of different specimens and different heatings of the same specimen. The final results were obtained by fitting a quadratic function in temperature to all the data. The function (standard deviation = 0.2%) that represents the electrical resistivity results for molybdenum in the temperature range 1500 to 2650 K is

$$\rho = -6.7083 + 2.8949 \times 10^{-2}\, T + 5.2985 \times 10^{-7}\, T^2 \tag{1}$$

where T is in K and ρ is in $\mu\Omega \cdot$ cm. The electrical resistivity of molybdenum computed using Eq. (1) is given in Table I. The deviations of the data for the different specimens and heatings from the values calculated from Eq. (1) are shown in Fig. 1. All the results reported in this paper are based on the International Practical Temperature Scale of 1968 [5]. Electrical resistivity values are based on the room temperature dimensions of the specimen.

The results of the electrical resistivity measurements performed at the ambient temperature (near 10°C) during the course of the experimental work are presented in Fig. 2. The data are fitted by a linear function in temperature using the least squares method, giving equal weight to each point. The standard deviation of an individual point from the smooth function is 0.1%. The temperature coefficient of the electrical resistivity of molybdenum (the slope of the linear function) at the ambient temperature is 0.017 $\mu\Omega \cdot \mathrm{cm} \cdot \mathrm{K}^{-1}$, which corresponds to a value of 0.32% per degree.

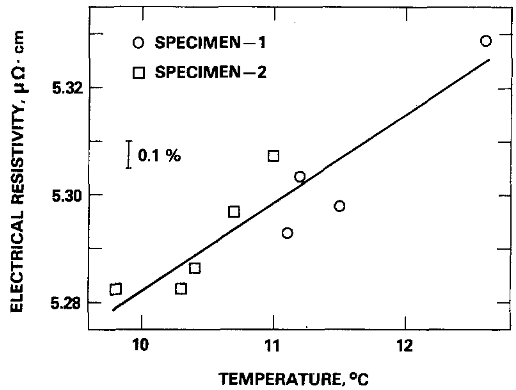

Fig. 2. Electrical resistivity of molybdenum near ambient temperature.

5. ESTIMATE OF ERRORS

The details for estimating the errors in measured and computed quantities in dynamic experiments with the present measurement system are given in an earlier publication [3]. In the present work, the specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publication. It may be seen from the results summarized in Table II that the imprecision[3] of the electrical resistivity measurements is 0.2%, and that the inaccuracy[4] in the results is estimated to be not more than 1%.

The effect of the specimen heating rate on the electrical resistivity measurements was studied by performing seven experiments on specimen 2 at different heating rates ranging from about 1000 to 8000 $K \cdot s^{-1}$. The results at 2000 K obtained from these experiments (Fig. 3) show that the measurements corresponding to heating rates lower than about 4000 $K \cdot s^{-1}$ were dependent on heating rate, while those higher than about 4000 $K \cdot s^{-1}$ were relatively insensitive to the changes in heating rate. The experiments (outside of the above seven) conducted in the present work correspond to heating rates in the range 5500 to 6700 $K \cdot s^{-1}$ (at 2000 K). The maximum difference in the electrical resistivity corresponding to these heating rates is less than 0.02%, which is less than the imprecision of the measurements.

[3]Imprecision refers to the standard deviation of an individual point as computed from the difference between the measured value and that from the smooth function obtained by the least squares method.

[4]Inaccuracy refers to the estimated total error (random, at one σ confidence level, and systematic).

Table II. Summary of the Error Analysis[a]

Quantity	Imprecision	Inaccuracy
Temperature (at 2000 K)	0.3 K	5 K
Current	0.03 %	0.1 %
Voltage	0.04 %	0.1 %
Electrical resistivity	0.2 %	1 %

[a]Definitions of the terms are given in the text.

When a specimen heats at a low rate, temperature gradients are established in the axial direction due to heat conduction from the specimen to the clamps. This lowers the effective temperature of the specimen with respect to its midpoint value. As a result, the measured electrical resistivity is lower than that under conditions of uniform temperature. Thus the effect of low specimen heating rates is low values for electrical resistivity, as is seen in Fig. 3.

6. DISCUSSION

The close agreement (within 0.1%) of electrical resistivity measurements at the ambient temperature several times during the course of the experimen-

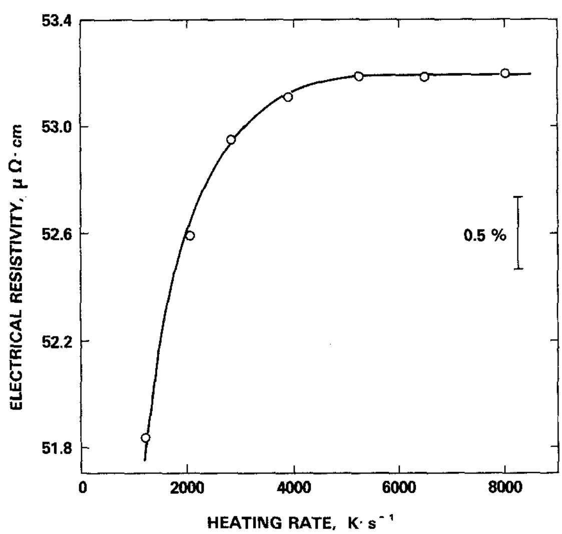

Fig. 3. Dependence of measured electrical resistivity (at 2000 K) of molybdenum on specimen heating rate.

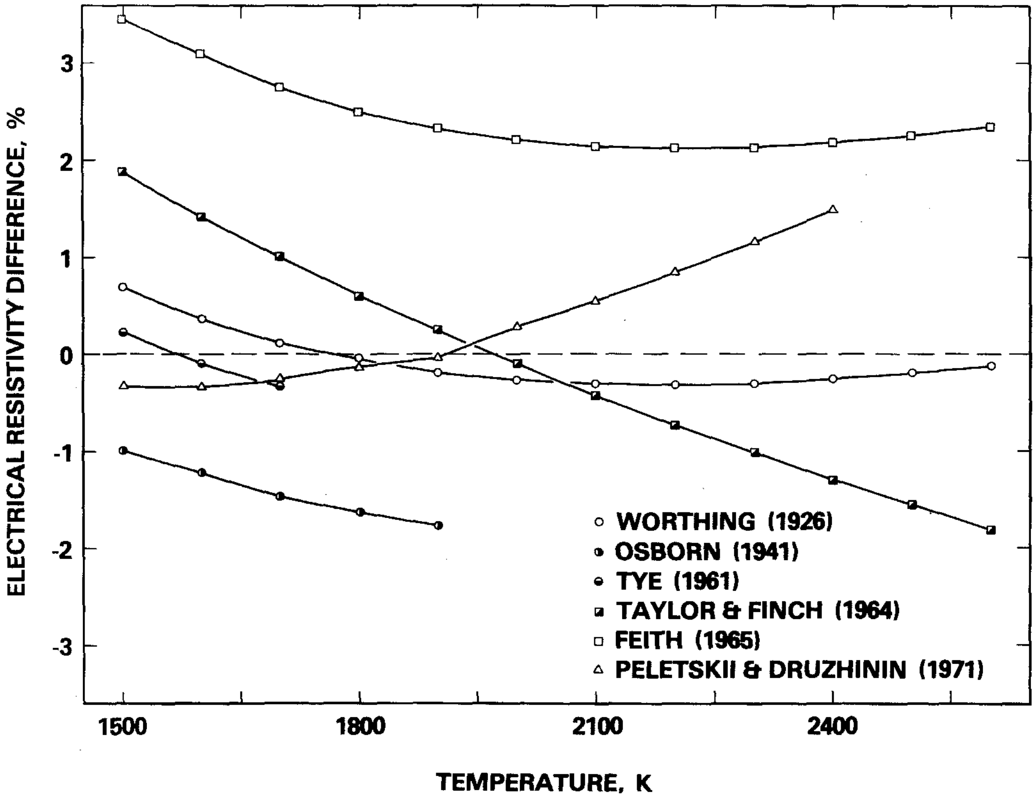

Fig. 4. Difference of electrical resistivity results reported in the literature from those of the present work. The "zero" line represents the present work results according to Eq. (1).

Table III. Electrical Resistivity Difference[a]

Investigator	Year	Temperature (K) 1,500	2,000	2,500
Worthing [6]	1926	0.7	−0.3	−0.2
Osborn [7]	1941	−1.0		
Tye [8]	1961	0.2		
Taylor and Finch [9]	1964	1.9	−0.1	−1.6
Feith [10]	1965	3.6	2.2	2.3
Peletskii and Druzhinin [11]	1971	−0.3	0.3	

[a]Previous literature values minus present values in percent.

tal work indicates that no significant irreversible structural and/or chemical changes had taken place in the specimens as the result of their heating to high temperatures. This is also substantiated by the reproducibility (within 0.1%) of the high temperature results on specimen 1 between first and second heating.

A comparison of the literature results with those of the present work is given graphically in Fig. 4 and for selected temperatures in Table III.[5] It may be seen that all the literature results fall in a bandwidth of about 5%. The best agreement of the present work values are with those of Worthing [6]: about ±0.5% over the range 1500 to 2650 K. The agreement with the results of Peletskii and Druzhinin [11] is within ±0.3% up to about 2000 K; however, the difference increases to about 1.5% at 2400 K. Estimates of errors by the authors in papers cited in Table III lead to an estimate of inaccuracy in previously reported electrical resistivity of approximately 1 to 3% in the

[5]In the comparison, all the electrical resistivity values are based on the room temperature dimensions of the specimens.

Table IV. Temperature Coefficient of Electrical Resistivity ($n\Omega \cdot cm \cdot K^{-1}$) of Molybdenum[a]

Investigator	Temperature (K) 1,000	1,500	2,000	2,500
Worthing [6]	28.0	29.3	30.7	32.0
Osborn [7]		29.0		
Tye [8]	26.7	29.1		
Taylor and Finch [9]	27.0	30.0	32.0	33.2
Feith [10]	28.0	29.6	31.3	32.9
Peletskii and Druzhinin [11]		30.2	32.5	
Present work		30.5	31.1	31.6

[a]Obtained from the electrical resistivity data reported by the investigators.

Table V. Electrical Resistivity of Molybdenum at 293 K Reported in the Literature

Investigator	Year	Purity (%)	Resistivity ($\mu\Omega \cdot$ cm)
Worthing [6]	1926		5.62
White and Woods [12]	1959		5.27[a]
Tye [8]	1961		5.65
Feith [10]	1965		5.86
Peletskii and Druzhinin [11]	1971	99.98	5.37[b]
Peletskii and Druzhinin [11]	1971	99.98	5.52[c]
Present Work[d]		99.95	5.45

[a] Ideal resistivity.
[b] For single crystal specimen.
[c] For polycrystalline specimen.
[d] Extrapolated from measurements in the range 283 to 285 K based on the value of 0.017 $\mu\Omega \cdot$ cm $\cdot$ K^{-1} for the temperature coefficient of the electrical resistivity of molybdenum.

temperature range considered. The agreement between data of various authors is within the combined estimated errors in all cases. In a recent paper, Peletskii [13] reviewed the data on the electrical resistivity of molybdenum reported in the literature, and presented "recommended values" in the range 300 to 2800 K. The values of the present work are somewhat lower than those given by Peletskii: by 0.1% at 1500 K, by 0.2% at 2000 K, and by 2% at 2600 K.

The temperature coefficient of electrical resistivity reported in the literature is given in Table IV. As may be seen, in all cases, the coefficient shows an increase with temperature. The value of the coefficient is in the range 29.0 to 30.5 n$\Omega \cdot$ cm $\cdot$ K^{-1} at 1500 K, and is in the range 31.6 to 33.2 n$\Omega \cdot$ cm $\cdot$ K^{-1} at 2500 K. The present value of the electrical resistivity of molybdenum at 293 K as well as the values reported in the literature are given in Table V.

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APPENDIX: TABLE A1

Table A1. Experimental Results on Electrical Resistivity of Molybdenum[a]

		Specimen 1					
		First heating		Second heating		Specimen 2	
Range	Temp. (K)	ρ	$\Delta\rho$	ρ	$\Delta\rho$	ρ	$\Delta\rho$
I	1,500	38.045	+0.36	38.107	+0.52	38.009	+0.26
	1,550	39.516	+0.20	39.564	+0.32	39.459	+0.06
	1,600	40.996	+0.07	41.052	+0.21	40.925	−0.10
	1,650	42.490	−0.03	42.562	+0.14	42.419	−0.19
II	1,650	42.516	+0.04	42.559	+0.14	42.423	−0.18
	1,700	44.025	−0.03	44.059	+0.05	43.919	−0.27
	1,750	45.554	−0.05	45.575	0.00	45.431	−0.32
	1,800	47.095	−0.05	47.110	−0.02	46.959	−0.34
III	1,800	47.077	−0.08	47.079	−0.08	46.944	−0.37
	1,850	48.619	−0.09	48.622	−0.08	48.477	−0.38
	1,900	50.159	−0.10	50.173	−0.07	50.027	−0.36
	1,950	51.706	−0.10	51.734	−0.05	51.592	−0.32
	2,000	53.273	−0.07	53.310	0.00	53.174	−0.25
IV	2,000	53.373	+0.12	53.301	−0.02	53.176	−0.25
	2,050	54.912	+0.09	54.875	+0.02	54.746	−0.22
	2,100	56.468	+0.08	56.449	+0.05	56.336	−0.15
	2,150	58.036	+0.09	58.025	+0.07	57.939	−0.07
	2,200	59.611	+0.11	59.604	+0.10	59.548	0.00
	2,250	61.190	+0.13	61.187	+0.13	61.152	+0.07
V	2,250	61.211	+0.16	61.260	+0.24	61.186	+0.12
	2,300	62.785	+0.17	62.818	+0.22	62.777	+0.16
	2,350	64.350	+0.16	64.378	+0.20	64.351	+0.16
	2,400	65.907	+0.13	65.936	+0.17	65.911	+0.13
	2,450	67.455	+0.08	67.488	+0.13	67.458	+0.09
	2,500	68.993	+0.02	69.032	+0.08	68.994	+0.02
	2,550	70.521	−0.05	70.563	+0.01	70.521	−0.05
	2,600	72.038	−0.14	72.077	−0.09	72.040	−0.14
	2,650	73.546	−0.25	73.567	−0.22	73.555	−0.24

[a]Electrical resistivity (ρ) is in $\mu\Omega \cdot$ cm. The quantity $\Delta\rho$ is the percentage deviation of the individual results from the smooth function represented by Eq. (1) in the text.