UCID - 16485

This is an informal report intended primarily for internal or limited external distribution. The opinions and conclusions stated are those of the author and may or may not be those of the laboratory.

MASTER

LAWRENCE LIVERMORE LABORATORY

University of California/Livermore, California

DEVELOPMENT OF EQUIPMENT FOR HIGH TEMPERATURE
RESISTIVITY MEASUREMENTS

S. Slivinsky

May 6, 1974

Prepared for U. S. Atomic Energy Commission under contract no. W-7405-Eng-48

NOTICE

Report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

GG

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DEVELOPMENT OF EQUIPMENT FOR HIGH TEMPERATURE RESISTIVITY MEASUREMENTS

ABSTRACT

Apparatus has been developed to measure resistivity accurately as a function of temperature at high temperatures for metals and alloys. The resistivity of 0.254-mm diam tantalum wire was measured between the temperatures of $1165\text{ K} \pm 7\text{ K}$ and $2862\text{ K} \pm 23\text{ K}$. Temperatures were measured with an automatic optical pyrometer, a two-color ratio pyrometer, and a tungsten-rhenium thermocouple. The temperature observations were made under simulated blackbody conditions. The good agreement with data from other experimenters indicates that the apparatus performs satisfactorily.

INTRODUCTION

To use a previously-developed ac technique for measuring heat capacities at high temperatures,¹ we must know the slope of the resistivity-temperature profile. This knowledge is necessary to obtain heat capacities from the data obtained with the ac technique. Resistivity as a function of temperature for refractory metals has been measured by a number of experimenters in a variety of ways.²⁻¹¹ In general their results disagree by a few percent to as much as 50%. The lack of agreement is more evident at high temperatures (temperatures that are two-thirds the absolute melting temperature and greater). We therefore decided to develop equipment that would accurately measure the resistivity of metals and alloys as a function of temperature at high temperatures. With accurate resistivity-temperature profiles and heat capacity measurements, we might then be able to obtain the energy of formation and concentration of vacancies at very high temperatures (near melting), and we could use the method on refractory metals and metallic alloys drawn into thin wires.

We developed and tested equipment for these measurements. We used tantalum as the test material because it is a refractory metal of reasonable

cost with a melting point of 3000°C and is readily available in wires of high purity. It is ductile at room temperature and is therefore easy to work. Resistivity data as a function of temperature exist with differences of a few percent,²⁻⁶ but data at temperatures exceeding 2800 K are sparse. We felt that if we could establish a reliable procedure using tantalum, it would give us the needed information to make heat capacity measurements, and the procedure could be used with confidence on alloys and on metals whose resistivities are less well known.

EQUIPMENT AND SAMPLES

Furnace and Vacuum System

We use a Brew* high temperature, high vacuum furnace, rated for 3000°C at 0.1 mPa. The furnace element is a single-phase tungsten mesh tube, split along two sides almost the entire length, with electrical connections at one end. It is 165 mm long by 51 mm in diameter. Seven cylindrical tungsten heat shields surround the element, with heat shield packets of seven tungsten disks each at the top and the bottom. The heat shields and heating element are enclosed in a water-cooled copper cylinder sealed at one end and closed at the other by inserting a copper disk to which the upper heat shield packet is attached. The outer vacuum envelope consists of a stainless steel container with ports for access to the furnace. Two of these ports contain ionization gauges, one for use during furnace operation and the other for backup. Another port provides access for backfilling the system with nitrogen before opening it to air. The nitrogen is admitted through a pressure release valve that protects the furnace from becoming over-pressurized. A Vacion pump is attached to the furnace through a fourth port and four Varian VacSorb pumps are attached

* Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Atomic Energy Commission to the exclusion of others that may be suitable.

through a fifth via a vacuum sealed shutoff valve. All temperature readings are taken from above, through a pyrex viewing port.

The temperature in the furnace is controlled with a feedback control system that maintains a constant input power to the tungsten heating element. As the power is manually set by the furnace operator, the controller senses the power to the heating element through a Hall effect transducer. The transducer produces a dc signal proportional to the product of the voltage and current to the heating element. The output signal from the transducer is compared to a reference voltage that is determined by voltage division with a series of high-stability precision resistors and a potentiometer. The resultant error signal is amplified and fed to a 20-kW silicon controlled rectifier pack (SCR). The output from the SCR is fed to a 30-kW stepdown transformer from which the 0- to 1000-A output passes through water-cooled vacuum feedthroughs to the heating element. For any given resistor and potentiometer setting the input power, and therefore the temperature, is held constant.

The vacuum system operates in the following manner: an air aspirator pump produces a pressure of 74 kPa in the system. Two VacSorb pumps are cooled in containers of liquid nitrogen and within a few seconds provide a pressure of 1.3 to 2 mPa. Once a pressure of 1.3 mPa is reached, it is possible to start the Vacion pump to produce a pressure of 10 μ Pa range in ~1 hr. Overnight operation at room temperature achieves pressures lower than 1.3 μ Pa. During the experiments on tantalum wire, the temperature was increased slowly so that the pressure was kept in the 1 μ Pa range as long as possible. Then the pressure was allowed to rise through the 10 μ Pa range slowly as the power was increased. We took no data when the pressure exceeded ~0.26 mPa.

Resistance Measuring Apparatus

We measured sample resistance directly with a Dana Digital Voltmeter (DVM) Model 4800 using the conventional four-wire arrangement. The voltage and current leads from the DVM to the sample were connected by copper

clamps to the copper water leads leading into the furnace. The DVM provides a 10.1 mA current on a fullscale resistance range of 10 Ω with an advertised accuracy of $\pm 0.005\%$ of the reading $\pm 0.005\%$ of the fullscale value at room temperature. For a reading of 2.000 Ω , this gives an accuracy of $\pm 0.03\%$. Our measurements with high precision resistors indicate that a 0.1% error on a scale of 10 Ω (the range used for all measurements) is a more realistic error estimate.

We made temperature readings with a Pyro-Automatic Optical Pyrometer, a Williamson Infrared Radiometer, and a tungsten-rhenium thermocouple.¹² The optical pyrometer was calibrated against an NBS blackbody ribbon filament lamp. The lamp uncertainties are $\pm 5^\circ$ at 800°C, $\pm 3^\circ$ at 1000°C, and $\pm 7^\circ$ at 2300°C. The pyrometer indicated 12° higher than the lamp at 800°C, 5° higher at 1100°C, and 22° higher at 2300°C.

The temperature indicated by the pyrometer, called the monochromatic brightness temperature, is lower than the true temperature if the object sighted on is not a blackbody. The true temperature is determined from the brightness temperature when the emissivity is known and is given¹³ by:

$$\frac{1}{T_T} = \frac{1}{T_b} + \frac{\lambda}{C_2} \ln \epsilon(\lambda, T_T),$$

where

λ = effective wavelength of the pyrometer (650 nm)

$C_2 = 14.38 \text{ mm} \cdot \text{K}$

$\epsilon(\lambda, T_T)$ = emissivity of the source at wavelength λ and temperature T_T

T_T = true temperature in K

T_b = brightness temperature in K.

The Williamson Infrared Radiometer is a two-color ratio pyrometer that obtains the ratio of the energies emitted by the sample surface at two different wavelengths. From Wien's equation for radiation emitted by an incandescent body,¹³ the ratio can be written as

$$R = \left(\frac{\epsilon_{\lambda_1}}{\epsilon_{\lambda_2}} \right) \left(\frac{\lambda_2}{\lambda_1} \right)^5 e^{-C_2 \left(\frac{\lambda_2 - \lambda_1}{\lambda_2 \lambda_1} \right) \frac{1}{T_R}}.$$

where

$\epsilon_{\lambda_1}, \epsilon_{\lambda_2}$ = emissivities at wavelengths 1 and 2

λ_1, λ_2 = 645 nm and 546 nm

C_2 = 14.38 mm·K

T_R = ratio temperature in K.

With the ratio pyrometer, the dependence on emissivity at a selected wavelength is replaced by a dependence on the emissivity ratio for the two wavelengths of operation. If the emissivity is wavelength-independent, the material is a gray body and the ratio temperature is identically equal to the true temperature. If the emissivity is one, the material is a blackbody and the ratio temperature is identically equal to the true temperature. Since the emissivity of most surfaces is a slowly varying function of wavelength, the emissivity ratio should still be close to one even if the material is not a gray body. This means the ratio temperature would be close to the true temperature of the sample. Our experiment uses blackbody conditions, so the ratio temperature and the true temperature are equal.

The ratio pyrometer has additional advantages, since it takes the ratio of the amplitudes of two signals. Thus, temperature measurements are not affected by prism and window transmission (given no selective absorption), the size of the source, or the intensity of the radiation. The instrument is not subject to errors due to drift since an internal calibration standard can be used to correct for drift immediately. It is sensitive to target temperature changes of 1° and is unaffected by ambient temperatures up to ~28°C.

The ratio pyrometer was calibrated against the same NBS blackbody as the optical pyrometer. The ratio pyrometer indicated 3° lower than the lamp at 1170°C, 5° higher at 1550°C, and 23° lower at 2535°C.

The tungsten-rhenium thermocouple is composed of 0.254-mm diam W-3% Re and W-25% Re wires, insulated with BeO. The sheath material is tantalum and the completed thermocouple assembly is 1.57 mm in diameter. These thermocouple wires were chosen because of their ductility and strength and their high recrystallization temperature. The maximum usable temperature

is limited by the insulation to 2200°C, according to the manufacturer's specifications.

The thermocouple is used in conjunction with a Joseph Kaye Thermocouple Ice Point Reference Chamber and an emf balancing bridge. The ice point reference chamber is a thermoelectric refrigerator controlled by the expansion of water upon freezing. It maintains a 0°C reference junction to within 0.0009 mV, as measured using an ice bath. The emf was read at every temperature for which optical measurements were taken to provide additional confirmation of the temperature measurements. This procedure was continued until apparent breakdown of the thermocouple insulation occurred at ~1960°C, somewhat below the specifications.

Samples

For precision of measurement, we wanted to get as much sample wire as possible into the experimental setup. We tried several shapes for the sample wire, including single coils, both loosely and tightly wound, and double coils where the second coiling was made by winding single-coiled wire in the opposite direction. These shapes were rejected because they resulted in sagging and shorting when heated. We then tried a U-shape that avoided both sagging and shorting and still made it possible to get a reasonably long sample into the small space available in the experimental arrangement.

The samples were 0.254-mm diam tantalum wire. Two samples were used in the measurements. Sample 1 was 165 mm $\pm$ 2 mm long, with a mass of 0.14095 g $\pm$ 0.0005 g. Sample 2 was 205 mm $\pm$ 2 mm long and weighed 0.18638 g $\pm$ 0.0005 g. The sample material was of Marz grade (99.999% purity). The U-shaped sample was spot-welded at both ends to Marz grade 0.762-mm diam tantalum wire, 108 mm long. When the 0.762 mm wires were clamped into position, the entire length of 0.254-mm diam sample material was within the hot zone of the furnace. The two voltage probes were 0.0762-mm tantalum wires of 99.9% purity (Marz grade probes being too fragile). Each was

spot-welded to a 0.762-mm wire, mounted in the assembly, and knotted to the sample ~25 mm from its ends. Knotting the probes to the sample made it possible to sinter them to the sample cleanly in situ as the sample was annealed in the furnace.¹⁴ The positioning of the probes was such that the portion of the sample between the probes was a constant temperature within the precision of measurement.

During initial resistance measurements of sample 1, we found that the resistivity-temperature profile was not reproducible above 2600°C. As long as the temperature remained above this value, the resistance continued to increase. When the temperature was lowered below 2600°C, the resistance stabilized but was greater at this temperature than it had been before the excursion above 2600°C. The following day the resistance was higher at all temperatures than it had been before. At temperatures above 2600°C the resistance began steadily increasing again and we could obtain no steady resistance reading under constant temperature conditions. When we stopped the experiment and weighed the sample, we found a 3% weight loss, indicating evaporation of the sample. Analysis showed that the sample contained 1% by weight of tungsten, probably obtained by evaporation from the tungsten mesh heating element.

To remedy these problems and to insure blackbody conditions, we installed a tantalum buffer. The buffer was a cylinder 170 mm long and 30.2 mm in diameter, closed at the bottom. It had a 42.9-mm diam rim welded to the open end and attached to the upper heat shield packet. The mounted tantalum sample was completely surrounded by this tantalum buffer, as was the tantalum-sheathed tungsten-rhenium thermocouple when it was in use. The temperature was observed through a circular opening 6.35 mm in diameter. The buffer was 97% black, according to DeVos' blackbody calculations.¹⁵

Figure 1 shows the furnace and vacuum system and a schematic of the sample, buffer, and thermocouple assembled inside the furnace.

a. Furnace and vacuum

b. Sample, buffer, and thermocouple

Fig. 1. Measurement apparatus.

RESULTS AND ANALYSIS

Temperature-Power Relations

Figure 2 shows temperature vs relative power curves. The temperature readings for Fig. 2a were taken with the optical pyrometer before the introduction of the blackbody buffer. These temperatures have been corrected for emissivity according to the Handbook of Chemistry and Physics.¹⁶ Losses due to prism and window transmission have been taken into account. These losses are shown for approximately every 100°C in Table 1.

Table 1. Transmission losses through prism and window.

Brightness temperature (°C)	Loss (°C)	Brightness temperature (°C)	Loss (°C)
800	11	1700	44
900	15	1800	47
1000	18	1900	52
1100	21	2000	58
1200	25	2100	61
1300	28	2200	65
1400	33	2300	70
1500	35	2510	75
1600	40		

The temperature readings for Fig. 2b are those taken with the ratio pyrometer after installation of the buffer. During the time in which these measurements were taken, we obtained numbers using the optical pyrometer, the ratio pyrometer, and the tungsten-rhenium thermocouple. Sample temperatures with both pyrometers were taken under simulated blackbody conditions and the thermocouple junction was placed next to the sample, well within the heated zone.

On the first warmup, the thermocouple, the power, and the ratio pyrometer were monitored simultaneously. Power, temperature, and temperature differences from this warmup are listed in Table 2. Next, the power and the optical pyrometer were monitored simultaneously. Power, temperature

Fig. 2. Temperature-power relations.

Table 2. Power vs temperature for thermocouple ratio pyrometer.

A	V	Power (W)	Thermocouple		Ratio pyrometer temperature (°C)	ΔT
			mV	Calibrated temperature (°C)		
300	2.5	0.0750	23.920	1291	1313	+22
440	4.5	0.1980	28.046	1517	1528	+11
480	5.2	0.2496	29.627	1609	1617	+ 8
520	5.8	0.3016	31.083	1695	1709	+14
630	8.0	0.5040	35.072	1951	1961	+10

Table 3. Temperature vs power for optical and ratio pyrometers.

A	V	Power (W)	Optical pyrometer temperature (°C)	Corrected optical pyrometer temperature ^a (°C)	Ratio pyrometer temperature ^b (°C)	ΔT
495	8.4	0.4158	1825	1873	1876	+ 3
390	5.8	0.2262	1520	1556	1577	+21
420	6.5	0.2730	1600	1640	1658	+18
520	9.3	0.4836	1880	1930	1941	+11

^aCorrected for prism and mirror transmissions.

^bCalculated from Table 2.

measured by the optical pyrometer, and temperature corrected for window and prism losses are listed in Table 3. Then we made interpolations of power vs ratio pyrometer temperature from Table 2 data to determine calculated ratio pyrometer temperature values for the temperatures measured by the optical pyrometer. The differences in these two temperatures are also shown in Table 3. Temperature differences for all these readings vary between 3°C and 22°C. The agreement between the two pyrometers and the thermocouple seems quite satisfactory for this temperature range.

Resistivity vs Temperature, Sample 1

We calculated sample resistivity from the resistance measurements, using $\rho = RA/l$, where R is specific resistance, A is specimen area, and l

is specimen length. The errors in these numbers are 0.1% for the resistance, 0.1% for the cross-sectional area, and 2 mm for the length of the sample, which amounted to 1.2% for sample 1 and 0.98% for sample 2. The overall uncertainty in ρ is therefore about $\pm 1.2\%$ for sample 1 and about $\pm 1\%$ for sample 2.

Figure 3 plots the resistivity vs temperature data for sample 1; the data are shown in Table 4. Data were taken from $1165\text{ K} \pm 5\text{ K}$ to $2862\text{ K} \pm 23\text{ K}$. The temperatures plotted were observed with the optical pyrometer and corrected for emissivity and losses through the prism and window. The data were obtained over several passes through each temperature shown. The power and therefore the temperature was successively increased and then decreased, and we found the resistance measurements to be reproducible within the uncertainty of measurement. We allowed ample time for equilibrium to be established for all measurements, especially on the decreasing temperature observations.

Measurements on sample 1 were terminated when the resistivity vs temperature profile was no longer reproducible because of sample evaporation and tungsten heater evaporation. Examination of the sample showed no change in diameter and no creep. Because of the care taken to insure data repeatability, we regard the data from sample 1 as reliable until the appearance of the alteration of the resistance vs temperature profile.

Resistivity vs Temperature, Sample 2

Figure 4 shows the resistivity vs temperature data for sample 2; the data are shown in Table 5. The data were taken between the temperatures of $1621\text{ K} \pm 11\text{ K}$ and $2562\text{ K} \pm 23\text{ K}$. The temperature measurements plotted in Fig. 4 are those observed with the optical pyrometer and the ratio pyrometer. Data were also taken with the thermocouple. The ratio pyrometer was used over the entire temperature range and was the more convenient of the two pyrometers at high temperatures. Its protective window could be closed off almost entirely while waiting for equilibrium, so it could still be used to indicate whether or not the temperature was still changing.

Fig. 3. Resistivity vs temperature, sample 1.

Fig. 4. Resistivity vs temperature, sample 2.

Table 4. Sample 1 data.

T_b^a (°C)	T_b^a (K)	T_T^b (K)	$T_T^{c'}$ (K)	R (Ω)	ρ ($\mu\Omega \cdot m$)	T_b^a (°C)	T_b^a (K)	T_T^b (K)	$T_T^{c'}$ (K)	R (Ω)	ρ ($\mu\Omega \cdot m$)
982	1255	1315	1328	1.782	54.30	1340	1613	1713	1743	2.254	68.72
945	1218	1274	1290	1.793	54.67	1375	1648	1752	1783	2.296	70.00
1195	1468	1550	1575	2.094	63.85	1435	1708	1820	1853	2.374	72.38
1725	1998	2153	2198	2.764	84.27	1490	1763	1883	1918	2.446	75.58
1690	1963	2113	2157	2.717	82.84	1540	1813	1940	1977	2.512	76.59
1630	1903	2043	2084	2.643	80.58	1585	1858	1992	2030	2.571	78.39
1570	1843	1974	2012	2.560	78.05	1625	1898	2037	2078	2.626	80.07
1495	1768	1890	1925	2.465	75.16	1665	1938	2084	2126	2.687	81.93
1415	1688	1798	1831	2.355	71.80	835	1108	1154	1165	1.610	49.09
1315	1588	1685	1713	2.230	67.99	1720	1993	2148	2192	2.760	84.15
1180	1453	1533	1557	2.066	62.99	1750	2023	2182	2227	2.803	85.46
1130	1403	1478	1500	2.002	61.04	1830	2103	2276	2324	2.878	87.75
1225	1498	1583	1608	2.119	64.61	1910	2183	2370	2422	2.967	90.46
875	1148	1198	1212	1.663	50.70	1990	2263	2464	2521	3.050	92.99
885	1158	1208	1222	1.672	50.98	1020	1293	1356	1374	1.860	56.71
1050	1323	1389	1408	1.891	57.66	2065	2338	2554	2614	3.126	95.31
1115	1388	1461	1482	1.972	60.13	2130	2403	2631	2693	3.196	97.45
1165	1438	1517	1540	2.040	62.20	2190	2463	2703	2767	3.280	100.01
1215	1488	1572	1597	2.102	64.09	2225	2498	2746	2812	3.328	101.47
1260	1533	1623	1650	2.158	65.80	2265	2538	2794	2862	3.386	103.24
1300	1573	1168	1696	2.208	67.32						

^aBrightness temperature.

^bTrue temperature corrected for emissivity.

^cTrue temperature corrected for emissivity and prism and window transmission.

We terminated the experiments on sample 2 when the voltage leads ruptured, causing an open circuit indication on the DVM. Sample 2 was shattered when it was removed from the furnace. However, the stability of the resistance during the readings obtained at constant high temperature indicated that creep, evaporation, and contamination were not significant during the measurements on sample 2.

Table 5. Sample 2 Data.

Temperature (°C)	Temperature (K)	Temperature ^a (K)	R (Ω)	ρ ($\mu\Omega\cdot m$)	Temperature (°C)	Temperature (K)	Temperature ^a (K)	R (Ω)	ρ ($\mu\Omega\cdot m$)
1375 ^a	1648 ^a	1679 ^a	2.403	68.08	2079	2352	-	3.170	89.80
1430 ^a	1703 ^a	1736 ^a	2.472	70.02	2140	2413	-	3.231	91.53
1475 ^a	1748 ^a	1782 ^a	2.535	71.81	2173	2446	-	3.253	91.65
1520 ^a	1793 ^a	1829 ^a	2.591	73.40	2202	2475	-	3.270	92.63
1560 ^a	1833 ^a	1871 ^a	2.643	74.87	2229	2502	-	3.283	93.03
1348	1621	-	2.314	65.55	1737	2010	-	2.790	79.04
1471	1744	-	2.471	70.02	1837	2110	-	2.905	82.30
1566	1839	-	2.588	73.31	1917	2190	-	2.999	84.96
1654	1927	-	2.689	76.18	1990	2263	-	3.078	87.20
1726	1999	-	2.770	78.47	1729	2002	-	2.778	78.70
1854	2127	-	2.917	82.63	1812	2085	-	2.854	80.85
1879	2152	-	2.945	83.43	1927	2200	-	3.004	85.10
1900	2173	-	2.975	84.27	1997	2270	-	3.076	87.14
1924	2197	-	3.000	84.98	2061	2334	-	3.136	88.84
1943	2216	-	3.027	85.75	2115	2388	-	3.190	90.37
1962	2235	-	3.049	86.37	2147	2420	-	3.220	91.22
1984	2257	-	3.093	87.62	2179	2452	-	3.250	92.06
1792	2065	-	2.866	81.19	2210	2483	-	3.285	93.06
1850	2123	-	2.934	83.11	2239	2512	-	3.306	93.65
1910	2183	-	2.993	84.79	2268	2541	-	3.324	94.16
1956	2229	-	3.044	86.23	2289	2562	-	3.338	94.56
2044	2317	-	3.133	88.76					

^aTaken with the optical pyrometer.

Comparison with Data of Other Experimenters

Figure 5 compares sample 2 data with the results obtained between 1600 K and 2800 K by other experimenters.^{2-5,17,18} Their measurements agree with ours to within 2 to 3%. Agreement with the data of Cezairliyan et al.¹⁸ is virtually perfect up to 2400 K. Above 2400 K, our resistivity vs temperature slope differs from those of Malter and Langmuir² and Cezairliyan. We could not determine Langmuir's experimental procedure even after an extensive literature search. However, Cezairliyan's high temperatures were achieved through rapid resistive heating of samples by a single pulse of current, with the specimen heated from room temperature to a temperature close to melting in <1 sec. We did our work statically and allowed ample time for equilibrium to be established. We made no measurements until all numbers were constant for a period of 10 min. It is possible that the static measurements bring about a different crystal defect state than the pulse experiments, resulting in different values of the measured resistivity.

There is no immediate explanation for the apparent downward bend in the profile. The bend is reproducible and has been repeatedly observed for both samples while both raising and lowering the temperature. We checked pyrometer calibration very carefully in these high temperature runs, with a calibration accuracy in the vicinity of 2400 K of ± 15 K. The most probable causes of a resistance change at these high temperatures are creep, impurities, and evaporation, all of which would cause the resistivity to increase rather than to decrease.

CONCLUSION

We have plotted resistivity vs temperature of tantalum between the temperatures of $1165 \text{ K} \pm 5 \text{ K}$ and $2862 \text{ K} \pm 24 \text{ K}$. The data agree with those found by previous experimenters within 2 to 3%. In view of this agreement, we conclude that our equipment can make accurate measurements of electrical resistivity at high temperatures.

Fig. 5. Tantalum resistivity vs temperature plotted by various researchers.

We also believe that our equipment can be used to obtain data at temperatures >2862 K. A runaway power problem during one of the experiments showed that the furnace can achieve 3300 K. However, to meet equilibrium conditions and maintain good vacuum in the low 0.1 mPa range, the practicable temperature range is probably somewhat less. If we lower the hydrocarbon level in the furnace by heating and outgassing, we add 100 to 200 K to the practicable temperature. Since at 2862 K the return temperature of the cooling water is quite warm, temperatures greater than ~ 3060 K for 10 min or more at 0.1 mPa will probably not be possible. With a limitation of ~ 3000 K, metals and alloys with lower melting temperatures than tantalum appear to be good candidates for these resistivity and heat capacity measurement techniques.

ACKNOWLEDGMENTS

The author expresses thanks to R. J. Borg for the helpful suggestions and advice provided during the course of this work and to M. Guinan, R. Van Konynenburg, D. Y. F. Lai, T. Westfall, and T. Shell for their invaluable assistance.

REFERENCES

1. R.M. Finnilla, An AC Temperature Technique for Measuring the High Temperature Specific Heat of Metals, Lawrence Livermore Laboratory, Rept. UCRL-50962 (1970).
2. L. Malter and D.B. Langmuir, Phys. Rev. **55**, 743 (1939).
3. B.E. Neimark and L.K. Voronin, High Temp. USSR **6**, 999 (1968).
4. V.E. Peletskii and V.Ya. Voskresenskii, High Temp. USSR **4**, 329 (1966).
5. V.A. Petrov, V. Ya Chekhovskoi, and A.E. Sheindlin, High Temp. USSR **6**, 525 (1968).
6. R.E. Taylor and R.A. Finch, Less-Common Metals **6**, 283 (1964).
7. A. Cezairliyan, M.S. Morse, H.A. Berman, and C.W. Beckett, J. Res. Nat. Bur. Stand. **74A**, 65 (1970).
8. A. Cezairliyan, J.L. McClure, and C.W. Beckett, J. Res. Nat. Bur. Stand. **75A** (1971).
9. P. Chiotte, Rev. Sci. Instrum. **25**, 876 (1954).
10. R.L. Rudkin, W.J. Parker, and R.J. Jenkins, Temp. Meas. Contr. Sci. Ind. **3**, 523 (1962).
11. R. Syre, Handbook on the Properties of Niobium, Molybdenum, Tantalum, Tungsten and Some of Their Alloys, (National Technical Information Service, Springfield, Va., 1965).
12. I.E. Campbell, High Temperature Materials and Technology (John Wiley, New York, 1967).
13. H.J. Kostowskii and R.D. Lee, Theory and Methods of Optical Pyrometry, National Bureau of Standards Monograph 41 (1962).
14. H. Schultz, "Quenching of Vacancies in Tungsten," in Lattice Defects in Quenched Metals, R.M.J. Cotterill, et al, Eds. (Academic Press, New York, 1965), p. 761.
15. J.C. DeVos, Physica **20**, 669 (1954).
16. Handbook of Chemistry and Physics, 51st Edition (Chemical Rubber Company, 1970) p. E-235.

17. A.G. Worthing, Phys. Rev. 28, 190 (1926).
18. A. Cezairliyan, J.L. McClure, and C.W. Beckett, J. Res. Nat. Bur. Stand. 75A (1971).

DISTRIBUTION

LLL Internal Distribution

J.W. Frazer, L-404
A. Maimoni, L-503
R.J. Borg, L-503 (2)
R. Van Konynenburg, L-503
S.L. Murray, L-503
TID File, L-3 (30)

External Distribution

S.H. Slivinsky (10)
739 Joyce Street
Livermore, California 94550

Technical Information Center (2)
Oak Ridge, Tennessee

NOTICE

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights."