The electrical resistivity and thermal expansion of iridium at high temperatures

by James Joseph Halvorson

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE in Aerospace and Mechanical Engineering
Montana State University

Abstract:
The electrical resistivity and thermal expansion of iridium were measured in the range of 293-2275°K and 891—2221°C respectively.

The electrical resistivity of iridium was obtained by measuring the dc-voltage across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. The following equation represents the results: ρt (293-2275 K) = $19.702 \times 10^{-3} t - 2.586 \times 10^{-6} t^2 + 4.64 \times 10^{-9} t^3 - 1.08 \times 10^{-15} t^4$ for the electrical resistivity, ρt, expressed in microhm-cm and the temperature t expressed in °K. The results of the present study were compared with earlier studies, and an attempt was made to correlate the experimental data to theory presented by Gruneisen. Mean temperature coefficients of resistivity were calculated.

The linear thermal expansion of iridium was measured in the range of 891-2221°C by taking the experimental data directly by visual means. The following equation was found to fit selected earlier results and the present results oyer the temperature range of 30-2221°C: $100 (L_t-L_0)/L_0 = 616.7 \times 10^{-6} t + 151.9 \times 10^{-9} t^2 - 28.16 \times 10^{-12} t^3 + 14.63 \times 10^{-15} t^4$ for t expressed in °C. Thermal expansion coefficients and average coefficients of expansion were calculated.

Statement of Permission to Copy

In presenting this thesis in partial fulfillment of the requirements for an advanced degree at Montana State University, I agree that the Library shall make it freely available for inspection. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by my major professor, or, in his absence, by the Director of Libraries. It is understood that any copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Signature James Joseph Halvarson

Date 12/3/71

THE ELECTRICAL RESISTIVITY AND THERMAL EXPANSION OF IRIDIUM AT HIGH TEMPERATURES

by

JAMES JOSEPH HALVORSON

A thesis submitted to the Graduate Faculty in partial
fulfillment of the requirements for the degree

of

MASTER OF SCIENCE

in

Aerospace and Mechanical Engineering

Approved:

E. H. Bishop
Head, Major Department

R. C. Weimer
Chairman, Examining Committee

K. Goering
Graduate Dean

MONTANA STATE UNIVERSITY
Bozeman, Montana

December, 1971

ACKNOWLEDGEMENTS

The present study was undertaken as part of a research program funded by the United States Atomic Energy Commission; gratitude is expressed for the financial support.

Thanks are expressed to Dr. R. E. Powe for use of the computer subroutines used in the data analysis and curve fitting.

Thanks are also expressed to Dr. E. H. Bishop, Aerospace and Mechanical Engineering Department, and to Dr. J. P. Hanton, Electrical Engineering Department, for serving on the thesis committee.

I would like to express a special thanks to Dr. R. T. Wimber. Without his support and guidance, this report would not be possible.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

The electrical resistivity and thermal expansion of iridium were measured in the range of 293-2275°K and 891-2221°C respectively.

The electrical resistivity of iridium was obtained by measuring the dc-voltage across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. The following equation represents the results:

$$\rho_t\ (293\text{-}2275°K) = 19.702 \times 10^{-3}t - 2.586 \times 10^{-6}t^2 + 4.64 \times 10^{-9}t^3 - 1.08 \times 10^{-15}t^4$$

for the electrical resistivity, ρ_t, expressed in microhm-cm and the temperature t expressed in °K. The results of the present study were compared with earlier studies, and an attempt was made to correlate the experimental data to theory presented by Grüneisen. Mean temperature coefficients of resistivity were calculated.

The linear thermal expansion of iridium was measured in the range of 891-2221°C by taking the experimental data directly by visual means. The following equation was found to fit selected earlier results and the present results over the temperature range of 30-2221°C:

$$100\ \frac{L_t - L_o}{L_o} = 616.7 \times 10^{-6}t + 151.9 \times 10^{-9}t^2 - 28.16 \times 10^{-12}t^3 + 14.63 \times 10^{-15}t^4$$

for t expressed in °C. Thermal expansion coefficients and average coefficients of expansion were calculated.

PART I

THE ELECTRICAL RESISTIVITY OF IRIDIUM AT HIGH TEMPERATURES

Introduction

The electrical resistivity of iridium was studied by Powell, Tye, and Woodman[1] in the range of 100-500°K. Other available electrical resistivity data generally refer to temperatures below 500°K. The values given in the "Thermophysical Properties of High Temperature Solid Materials"[2] in graphical form in the range of 75-1593°K are discussed later. (Values were obtained from the work of Powell, et al.[3]) A need for additional resistivity data for iridium at higher temperatures prompted making the resistivity measurements presented in the present paper.

As the experimental data reported in the present paper were being obtained, results of a similar study made by Russian workers appeared in the technical literature. The electrical resistivity of iridium was studied by L'voy, Mal'ko and Nemchenko[4] in the range of 80-1900°K. Iridium samples were prepared by a powder metallurgy method (pressed billets were sintered in vacuum at a temperature of about 2500°K). Electrical resistivity data were obtained by the potentiometric method and are compared with the results of the present study.

Specimen Analysis

Commercially pure iridium wire, 0.635 mm (25 mils) in diameter was purchased from Engelhard Industries, Carteret, New Jersey. Results of a spectrographic analysis* of the wire are contained in Table I along with the results of a vacuum fusion analysis** for oxygen, hydrogen, and nitrogen. The total measured impurity content of the metal was seen to be 0.028%. Although analysis did not include all known elements, the iridium content was estimated to exceed 99.95%.

Experimental Method

The experimental equipment used in the present study was designed such that the electrical resistivity was obtained by measuring the dc-voltage drop across the specimen and the voltage drop across a precision resistor which was in series with the wire specimen. The ratio of voltages corresponded to a resistance ratio from which the electrical resistivity was calculated knowing the length and diameter of the specimen. Figure 1 is a schematic representation of the experimental equipment. A ceramic plug which contained the iridium wire specimen was made from a castable ZrO_2 cement. The wire specimen was electrically isolated from the ZrO_2 plug by passing the wire through ThO_2 thermocouple tubes, ThO_2 being used because of its high electrical resistivity at high temperatures. The section of the iridium wire specimen for which the resistivity was measured was of uniform temperature

*Performed at the Matthey Bishop Co., Malvern, Pennsylvania.

**Performed at the Magnflux Testing Laboratories, Los Angeles, California.

TABLE I ANALYSIS OF THE IRIDIUM WIRE

(Amounts of Impurity Elements Expressed in PPM)

Pt	15	Sb	<10	Fe	87	Sn	< 1
Pd	5	As	<10	Pb	< 1	Ti	6
Rh	54	Bi	< 1	Mg	5	Zn	<10
Ru	25	B	5	Mn	1	Ca	7
Os	< 3	Cd	< 1	Mo	1	O	30
Au	3	Cr	< 1	Ni	3	H	1
Ag	< 1	Co	< 1	Si	7	N	15
Al	9	Cu	2	Te	<10		

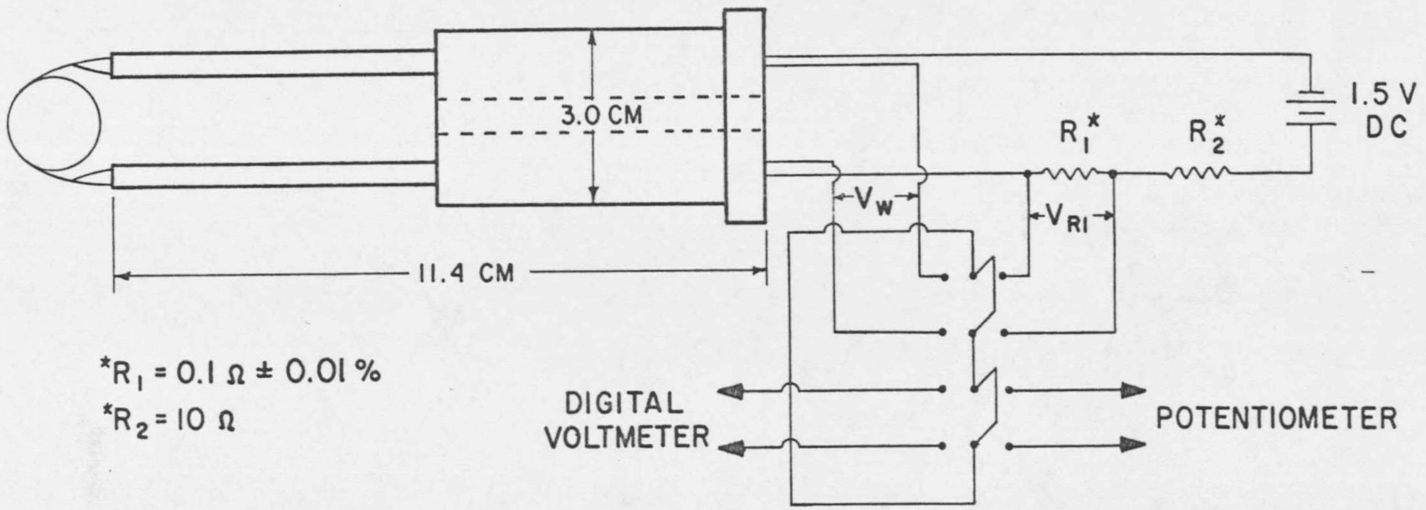

Figure 1. Schematic Representation of the Experimental Equipment

and was located a distance of one centimeter from the end of the ThO_2 thermocouple tubes. A ZrO_2 sight tube was also incorporated in the plug and thus allowed measuring the higher temperatures with a Pyro-Micro-Optical Pyrometer and the lower temperatures by insertion of a chromel-alumel thermocouple. Pyrometer calibration factors were obtained using a Milletron Pyrometer Calibration Standard containing a lamp calibrated by the General Electric Co. (with all measurements traceable to the National Bureau of Standards). The iridium wire specimen was connected in series with a 0.1Ω ± .01% precision resistor. The circuit was completed with a 10Ω resistor and 1.5 dc volt power supply. Both resistors were made from Manganin wire having a temperature coefficient of resistivity of 10 ppm/°C and thus were unaffected by the small changes in room temperature.

The wire specimen was prepared by fusion-welding* two 14-cm iridium lead wires to another piece of the iridium wire having a length of 34 cm. The room-temperature length of the reference section (7.96 cm) of the iridium wire was accurately determined from its weight, diameter, and a published[5] value for the density equal to 22.49 $g\text{-}cm^{-3}$ after the reference section was cut from the system at the conclusion of the series of resistivity measurements. The diameter of the wire was

*The fusion-welding process is described in Appendix A.

measured optically to ±1μ using a Gaertner Cathetometer, and the weight determined with a precision of ±0.1 mgm. Measurement of the wire diameter before and after the series of resistivity measurements yielded no detectable change. Copper lead wires were fusion-welded to the ends of iridium wires extending from the ends of the ThO_2 tubes; the copper-iridium thermal EMF over the range of 0-100°C was reported[6] to be only 1.1μv/°C. Differences in the temperatures of the four Ir-Cu junctions was minimized by directing a stream of air across the junctions which were in close proximity.

Prior to making the resistivity measurements, the reference section of the specimen was given a recrystallization anneal by heating in argon to approximately 2300°K for 60 minutes in the high-temperature furnace described later.

In preparation for the measurements at specimen temperatures in the range of 293-1241°K, the plug containing the wire specimen was placed in a type 54341-A Lindberg Hevi-Duty furnace having a maximum operating temperature of 1283°K. After sealing, the furnace was continuously flushed with argon to prevent oxidation of the specimen. The furnace temperature was controlled with a Variac sliding-contact autotransformer and was monitored with a digital millivoltmeter connected to a Platinel II thermocouple installed in the furnace. Continuous specimen temperature readings were provided by a Digitec dc millivoltmeter connected to a chromel-alumel thermocouple placed next to the iridium wire specimen. Both thermocouples had an ice-bath reference. After the furnace temperature had stabilized, the temperatures indicated by the separate thermocouples were nearly the same. The voltage drops across the reference section of the wire specimen (V_w) and the precision resistor (V_{R1}) were determined using a Honeywell model 2745 potentiometer with a precision of ±0.001 millivolts after preliminary values were obtained using a digital voltmeter (Hewlett Packard Model 3440A having a precision of ±0.01 millivolts). The use of the digital voltmeter (and two double-pole double-throw switches) allowed approximate voltage values to be measured quickly; subsequently the values were measured precisely using the potentiometer, thus allowing a rapid data-collecting procedure. The error due to a very slow drift in the current and voltage provided by the dc power supply was eliminated by alternately and repetitively measuring V_w and V_{R1}. Repeated measurements at the same temperature were reproducable to ±0.005 millivolts.

For the data taken at specimen temperatures in the range of 1585-2275°K, the plug containing the specimen was installed in a ZrO_2 refractory tube which was in turn located inside the tantalum heating element of a high-temperature furnace to which argon was fed. The argon was passed through a purifier containing calcium turnings held at approximately 925°K to remove any traces of oxygen and water vapor present. The purified argon was then introduced into the furnace.

A cast ZrO_2 cylinder and heat-reflector discs were placed in the refractory tube beneath the wire specimen. These discs completed an enclosure for the wire; when heated, this enclosure closely approached being a black-body cavity such that the pyrometer readings were assumed to correspond to true temperature. Temperature measurements were taken with a Pyro-Micro-Optical Pyrometer, which was sighted through the sight tube located in the plug (the pyrometer was calibrated by sighting through this same sight tube onto the calibration lamp mentioned earlier). Voltage measurements for specimen temperature exceeding 1585°K were made using only the digital voltmeter (thus, voltage drops in the range of 13-21 millivolts were measured with a precision of ±0.01 mv). Repeated measurements at the same temperature were reproduceable to ±0.05 mv.

The results of resistivity measurements at 77-273°K and the procedure employed in their determination are contained in Appendix B.

Results and Discussion

The electrical resistivity data obtained in the present study in the range of 293-2275°K are presented in Table II and Figure 2. All of the data tabulated by L'vov, et al[4], are also shown; their tabulated values are in good agreement with those of the present study, with maximum deviation being less than 5.2%. The data points presented graphically by L'vov, et al, at the higher temperatures were noticed to