



Natural convection high temperature oxidation of iridium
by Hal Gene Kraus

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE in Mechanical Engineering
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Abstract:

The experimental study of the natural convection oxidation of iridium was performed at temperatures of 1600, 1900, and 2200°C and pressures ranging- from 0.00132 to 1.32 atmospheres. A theoretical model describing the surface, recession rate was derived assuming that the rate was controlled by the diffusion of Ir, IrO₂, and IrO₃ through the gaseous boundary layer, adjacent to the iridium specimen. Average values for the standard state enthalpy and entropy changes for the formation of IrO_g and IrCh were calculated for the 1600 to 2200 °C temperature range. A value for the partial pressure of Ir at 2200°C was found. Correlation between experiment and the theoretical model was good with the overall mean % error of experimental rates from theoretically predicted rates being 13.3% for the 1600 to 2200°C range.

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Date May 18, 1973

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HAL GENE KRAUS

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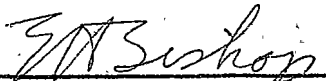
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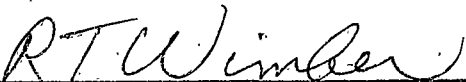
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TABLE OF CONTENTS

	Page
LIST OF TABLES	v
LIST OF FIGURES	vi
NOMENCLATURE	vii
ABSTRACT	xi
INTRODUCTION	1
REVIEW OF PAST WORK	2
THEORY	7
IRIDIUM ANALYSIS	8
EXPERIMENTAL EQUIPMENT AND PROCEDURE	8
EXPERIMENTAL RESULTS AND THEORETICAL RATE CALCULATIONS	17
DISCUSSION OF RESULTS	36
SUMMARY AND CONCLUSIONS	38
BIBLIOGRAPHY	40

LIST OF TABLES

Table	Page
I. DEPENDENCY OF RECESSION RATE ON OXYGEN PARTIAL PRESSURE . . .	6
II. SPECTROGRAPHIC ANALYSIS OF IRIDIUM	9
III. EXPERIMENTAL DATA	19
IV. OXYGEN PROPERTIES	22
V. SUMMARY OF ΔH_f° AND ΔS_f° VALUES OF PAST EXPERIMENTERS	28
VI. INITIALLY PREDICTED EQUILIBRIUM CONSTANTS	28
VII. OPTIMIZATION RESULTS - FINAL VALUES FOR EQUILIBRIUM CONSTANTS AND ACCOMODATION COEFFICIENT	31
VIII. FINAL CORRELATION DATA	34
IX. TYPICAL TRENDS FOR VOLATILE DIOXIDE AND TRIOXIDE SPECIES AS A FUNCTION OF TEMPERATURE	37

LIST OF FIGURES

Figure	Page
1. EXPERIMENTAL EQUIPMENT SCHEMATIC	11
2. SAMPLE PLOT FOR DETERMINATION OF EXPERIMENTAL SURFACE RECESSION RATE	18
3. PLOT OF EXPERIMENTAL AND THEORETICAL RECESSION RATES VERSUS OXYGEN PARTIAL PRESSURE	33

NOMENCLATURE

Symbols

A, Effective equivalent weight equal to 192.2 gms of iridium consumed per gm mole of oxide formed divided by the density of the iridium

α , Accomodation coefficient

β , Coefficient of thermal expansion - $1/T_f$, ($^{\circ}\text{K}$) $^{-1}$

C, Constant in diffusivity equation

CF, Correction factor for heat transfer coefficient due to cell enclosure

C_p , Constant pressure heat capacity of oxygen - cal/gm $^{\circ}\text{K}$

D, D_{av} , Average experimental wire diameter - cm

D_v , Molecular diffusivity - cm 2 /sec

ΔF_f° , Standard state free energy change - kcal/mole

ΔH_f° , Standard state enthalpy of formation - kcal/mole

ΔS_f° , Standard state entropy of formation - eu (kcal/mole $^{\circ}\text{K}$)

\mathcal{E}_{12} , Energy of molecular interaction - ergs

\mathcal{E}_1/K , Force constant for oxygen - $^{\circ}\text{K}$

\mathcal{E}_2/K , Force constant for oxide specie - $^{\circ}\text{K}$

f, Friction factor

g, Gravitational acceleration - cm/sec 2

γ , Specific heat ratio of oxygen

G_M , Molal mass velocity - gm moles/sec cm 2

Gr, Grashof number - $g\beta(T_s - T_a)D^3/\mu^2$

h, Film heat transfer coefficient - cal/cm 2 sec $^{\circ}\text{K}$

Symbols

I_D , Collision integral for diffusion - function of $\bar{k}T_p/\epsilon_{12}$

j_D, j_H , Chilton - Colburn factors

k , Thermal conductivity of oxygen - cal/cm sec °K

\bar{k} , Boltzmann constant - 1.38×10^{-6} ergs/°K

k_c , Mass transfer coefficient - gm moles/(sec cm²)(gm mole/cm³)

k_G , Gas-film mass transfer coefficient for diffusion of oxide specie through the boundary layer - gm mole/sec cm² atmos

K , Equilibrium constant

λ_m , Mean free path of oxygen molecules - cm

Le , Lewis number - Pr/Sc or $C_p \rho D/k$

M_1 , Molecular weight of oxygen

M_2 , Molecular weight of oxide specie

μ , Viscosity of oxygen - cp (centipoise or gm/cm sec)

μ_t , Viscosity of oxygen - p (poise)

n , Exponent of P_{O_2} where $x_e \propto (P_{O_2})^n$

Nu , Nusselt number - hD/k

Nu^* , Fictitious Nusselt number

P , Total pressure - atmos

P_{BM}, P_{O_2} , Partial pressure of oxygen - atmos

P_{Ir} , Partial pressure of iridium - atmos

P_t , Partial pressure of oxygen - torr

Pr , Prandtl number - $C_p \mu/k$

Symbols

$(r_o)_1$, $1.18(V_o)_1^{1/3}$ - collision diameter for oxygen molecule/oxygen molecule collision - angstroms

$(r_o)_2$, $1.18(V_o)_2^{1/3}$ - collision diameter for oxide molecule/oxide molecule collision - angstroms

r_{12} , $((r_o)_1 + (r_o)_2)/2$ - collision diameter for oxygen molecule/oxide molecule collision - angstroms

ρ , Density of oxygen - gm/cm³

ρ_M , Density of iridium - gm/cm³

R, Universal Gas Law Constant - 81.7 cm³ atmos/gm mole °K or 1.987 cal/gm mole °K

Re, Rayleigh number - GrPr

Sc, Schmidt number - $\mu/\rho D_v$

T_a , Ambient cell temperature - °C

T_f , Experimental film temperature - °K

T_k , Experimental wire temperature - °K

T_m , Melting temperature for oxides - °K

T_s , Experimental wire temperature - °C

U_{av} , Mean gas velocity - cm/sec

$(V_o)_1$, Molal volume of oxygen - cm³/gm mole

$(V_o)_2$, Molal volume of oxide specie - cm³/gm mole

\dot{x}_e , Experimental surface recession rate - cm/sec

\dot{x}_{se} , Standardized experimental surface recession rate at $D = 0.588$ cm - cm/sec

x

Symbols

\dot{x}_{st} , Standardized theoretical surface recession rate at $D = 0.588$ cm - cm/sec

\dot{x}_t , Theoretical surface recession rate - cm/sec

ABSTRACT

The experimental study of the natural convection oxidation of iridium was performed at temperatures of 1600, 1900, and 2200 °C and pressures ranging from 0.00132 to 1.32 atmospheres. A theoretical model describing the surface recession rate was derived assuming that the rate was controlled by the diffusion of Ir, IrO₂, and IrO₃ through the gaseous boundary layer, adjacent to the iridium specimen. Average values for the standard state enthalpy and entropy changes for the formation of IrO₂ and IrO₃ were calculated for the 1600 to 2200 °C temperature range. A value for the partial pressure of Ir at 2200 °C was found. Correlation between experiment and the theoretical model was good with the overall mean % error of experimental rates from theoretically predicted rates being 13.3% for the 1600 to 2200 °C range.

INTRODUCTION

Iridium is one of the platinum group metals. It is the only elemental metal known to man possessing a high enough melting point and a low enough oxidation rate to be used unprotected in air for an extended period of time in the 2000 to 2300 °C range. A thin oxide film is formed on its surface when iridium is subjected to temperatures from 600 to 1000 °C in an oxygen containing gas. When the temperature of the iridium exceeds 1000 °C it forms volatile oxides, and the oxidation rate becomes linear with time, leaving the surface shiny.

Iridium is considered to be the most corrosion resistant metal when it is compared with all other metals over a broad range of temperatures and environments. Numerous acids, salt solutions, fused salts, molten metals, and molten metal oxides are among the corrosive environments considered.

Iridium has been used for standardized high temperature thermocouples, for heating elements in high temperature furnaces, and for protective coatings for less expensive high temperature structural materials such as tungsten and tantalum base alloys. The most recent research on iridium oxidation has shown the potential applications for the leading edge of spacecraft, for components in nuclear reactors, and for parts in rocket and turbine engines.

Being a platinum group metal, iridium is expensive. This limits its use as a primary material in structural applications, even though iridium has reasonably good strength properties.

Among other properties, iridium has very low emittance and very high density. The low emittance causes problems when iridium is used to any extent on spacecraft because the primary means of heat dissipation on the nose of spacecraft re-entering the earth's atmosphere is by radiation. Where weight is a constraint, the 22.57 gm/cm^3 density of iridium (at room temperature) greatly restricts its application. In fact, iridium is either the most or the next to the most dense element known to man. (Controversy exists on whether iridium or osmium is the most dense due to the inconsistencies of density measurements based on the lattice structure and actual physical measurements. Actual measurements reveal iridium to be the most dense.)

REVIEW OF PAST WORK

A short review of the recent but relevant work done on the oxidation of iridium is in what follows. From this review, conclusions of a general nature were made which served as a starting basis for the work of this report.

The composition of the products of iridium oxidation and the thermodynamics involved have been investigated by several people. Cordfunke and Meyer¹ passed oxygen, or oxygen-nitrogen mixtures, over a boat shaped specimen containing iridium in the range of 1169 to 1462 °C. They concluded that the main volatile oxide formed was IrO_3 .

Holburn, Henning, and Austin² self resistance heated a piece of thin iridium foil to 1670 °C over which they passed oxygen, air, or nitrogen con-

taining 1.77% oxygen. They also concluded that IrO_3 was the main gaseous oxide specie formed.

Norman et al.,⁴ performed a sequence of mass spectrometric studies of oxidizing iridium. This was done by introducing oxygen into a Knudsen Cell in which an iridium specimen was placed and heated in the range of 1557 to 1760°C. The exiting gaseous species were then "bombarded" with a stream of electrons. They detected the presence of Ir^+ , IrO^+ , IrO_2^+ and IrO_3^+ ions, but concluded that IrO_2 and IrO_3 were the main volatile oxide species present in their work.

Various related kinetic studies have also been performed. Norman et al. also measured values of the standard state enthalpy and entropy changes for the formation of IrO_2 of 48.5 ± 0.5 kcal/mole and 3.9 ± 2.0 eu, and for IrO_3 5.5 ± 1.5 kcal/mole and -13.1 ± 2.5 eu in their respective orders.

Olivei⁵ performed some mass spectrometric studies also, in the range of 1627 to 2227°C, from which it was found that the standard state enthalpy and entropy of formation for IrO_3 were 6.0 kcal/mole and -9.0 eu respectively.

For the free entropy of formation of oxides of a general metal M, values of 20.6, 3.5 ± 2.1 , and -10 to -15 eu, as averages for volatile gaseous monoxide MO, dioxide MO_2 , and trioxide MO_3 species respectively, were indicated by Searcy.⁶

Phillips⁷ performed some kinetic studies of iridium oxidation in

air moved only by natural convection. The specimen was a 1/4 inch in diameter by 1/2 inch long iridium rod. The range of temperatures investigated was 650 to 1300 °C for which Phillips found an activation energy of 9.2 kcal/mole.

Krier and Jaffee⁸ heated an iridium specimen by means of a furnace through which slow moving air was passed in the range of 1000 to 1400 °C. They found a value of 16 kcal/mole for the activation energy.

Criscione⁹ passed oxygen or an oxygen-argon mixture parallel to a 1/2 inch diameter iridium rod having a hemispherically shaped end, in the range of 1150 to 1900 °C and at a pressure of 0.935 atmospheres. He measured surface recession rates for the hemispherical end and found an activation energy of 23 kcal/mole.

Work by Kuriakose and Margrave¹⁰ involved heating an iridium foil 0.002 inches thick suspended in a furnace for temperatures up to 1301 °C and resistance heating a 0.01 inch in diameter iridium wire inside a 1 inch diameter pyrex tube for higher temperatures. They passed oxygen or oxygen-helium mixtures through the tube parallel to the tube length at a total pressure of 1 atmosphere. For the iridium foil they found activation energies of 70 and 7.1 kcal/mole for temperatures below 1100 °C, and for temperatures between 1100 and 1300 °C respectively. Activation energies for the iridium wire were found to be 12.5 and 30-33 kcal/mole at 1150 to 1600 °C, and 1600 to 2217 °C respectively.

Among other dependencies, it has been found that the surface reces-

sion rate of a specimen depends upon the oxygen partial pressure to some power. Table I summarizes this dependency as found by various experimenters of which Tempero¹¹ is the only one not previously mentioned.

Except for the work recently done by Wimber and Tempero, no work has led to the characterization of the oxidation process which would allow taking into account the effects of varying temperature, oxygen partial pressure, flow rate past a specimen and specimen size. Wimber and Tempero resistance heated an iridium wire in the range of 1600 to 2200 °C. The wire was varied from 0.0635 to 0.0381 cm in diameter and oxygen was passed perpendicular to the wire. Among their investigations of describing the recession rate theoretically, they had some success in a model which considered the equilibrium diffusion of gaseous IrO_2 and IrO_3 through the boundary layer adjacent to the specimen.

In view of the mentioned past work on iridium oxidation, the following conclusions were arrived at.

1. IrO_2 and IrO_3 appear to be the main volatile oxide species formed above 1600 °C.
2. Values for the standard state enthalpy and entropy of these two major species are known at certain temperature levels, but are expected to vary with temperature.
3. Activation energies are expected to vary with specimen environment of oxygen, specimen configuration, and specimen temperature.
4. The surface recession rate, at least, depends upon the oxygen par-

Table I. DEPENDENCY OF RECESSION RATE ON P_{O_2}

$$x_e \propto (P_{O_2})^n$$

	Holburn et al	Criscione	Kuriakose & Margrave	Tempero
Iridium Specimen Environment	Foil - slow flowing	Rod - parallel flow	Foil & wire parallel flow	Wire - perpendicu- lar flow
Exponential Dependency - Temperature Range	n = 1.5 @ 1670 °C	n = 1.34 @ 1900 °C	Foil n = 1.1 @ 1181 °C Wire n = 1.2 @ 1717 °C	n = 0.234 @ 1600 to 2200 °C
Oxygen Partial Pressure Range	P_{O_2} = 0.017 to 1.00 atmos	P_{O_2} = 0.02 to 0.93 atmos	P_{O_2} = 0.02 to 1.00 atmos	P_{O_2} = 0.0046 to 1.30 atmos

tial pressure, and the specimen configuration and temperature.

THEORY

When iridium is oxidized above 1000°C it forms volatile oxide species, for which the associated rate is expected to be controlled by one or more of the following steps in the process:

1. Bulk transport of the oxygen bearing gas from the source to the general vicinity of the metal being oxidized.
2. Diffusion of the oxygen through the gaseous boundary layer adjacent to the surface of the metal.
3. Adsorption of the oxygen from the gas phase onto the surface of the metal.
4. Reaction on the metal surface including possible rearrangement and migration processes.
5. Desorption of the oxide(s) from the surface of the metal.
6. Diffusion of the oxide(s) through the gaseous boundary layer with possible further chemical reaction(s) within the boundary layer of one of the oxide species with the same or different oxide specie and/or oxygen.

A theoretical model was derived by Wimber¹², which takes into account the formation of IrO , IrO_2 , and IrO_3 at their equilibrium partial pressures on the inner surface of the boundary layer. The model also accounts for metal vaporization which occurs to an appreciable extent at high temperatures and low pressures. This model assumes that the surface recession

rate \dot{x}_t , is controlled by the rate of diffusion of these gaseous species through the gaseous boundary layer corresponding to the first part of step 6. and was expressed as,

$$\dot{x}_t = A(k_{G1}K_1P_{O_2}^{1/2} + k_{G2}K_2P_{O_2} + k_{G3}K_3P_{O_2}^{3/2} + k_{GM}K_M), \quad (1)$$

where the subscripts 1,2,3, and M refer to IrO , IrO_2 , IrO_3 , and Ir respectively, and:

A = Effective equivalent weight equal to 192.2 gms of iridium consumed per gm mole of oxide formed divided by the density of Ir,

k_G = Gas-film mass transfer coefficient - gm mole/sec cm^2 atmos,

K = Equilibrium constant,

P_{O_2} = Partial pressure of oxygen - atmos.

Validity of equation (1) was tested by comparison of experimental data taken in the natural convection area, to results predicted by this model.

IRIDIUM ANALYSIS

The oxidation studies in natural convection were conducted using two different lots of commercially pure iridium, purchased from Engelhard Industries, Cateret, New Jersey. A spectrographic analysis of this iridium was requested of Engelhard Industries Division. These analyses are shown in Table II. The results of these two lots of iridium were compared experimentally.

EXPERIMENTAL EQUIPMENT AND PROCEDURE

Figure 1 is a schematic representation of the equipment used. An iridium specimen was placed horizontally between two electrodes in a cell

Table II. SPECTROGRAPHIC ANALYSIS OF IRIDIUM*

Impurity	Lot #1 diameter 25 mils	Lot #2 diameter 20 mils
Pt	99	<0.5
Rh	20	38.0
Pd	10	11.0
Ru	166	1.5
Au	<1	<0.5
Ag	50	-
Pb	<5	<0.5
Sn	5	<0.5
Zn	<10	0.41
Fe	170	64
Cu	96	17
Si	43	6.1
Mg	41	1.1
Ca	78	0.61
Al	14	35
Ni	10	1.1
Cr	31	0.84
Mn	10	0.69
Sb	<10	<0.5
B	<50	0.0032
Co	100	1.8
As	<10	0.066
Bi	<1	-
Cd	<1	-
W	0.6%	710
N	50	0.24
H	4	-
O	70	8.3
Ta	-	46
In	-	0.26
Zr	-	1.1
Ga	-	0.20
V	-	0.096
Ti	-	0.49
K	-	0.67
Cl	-	0.59
S	-	0.57

Table II. (Continued)

P	-	0.81
Na	-	2.2
F	-	0.27
C	-	15.0
Li	-	0.00072

* Impurities are given in parts per million unless otherwise specified.

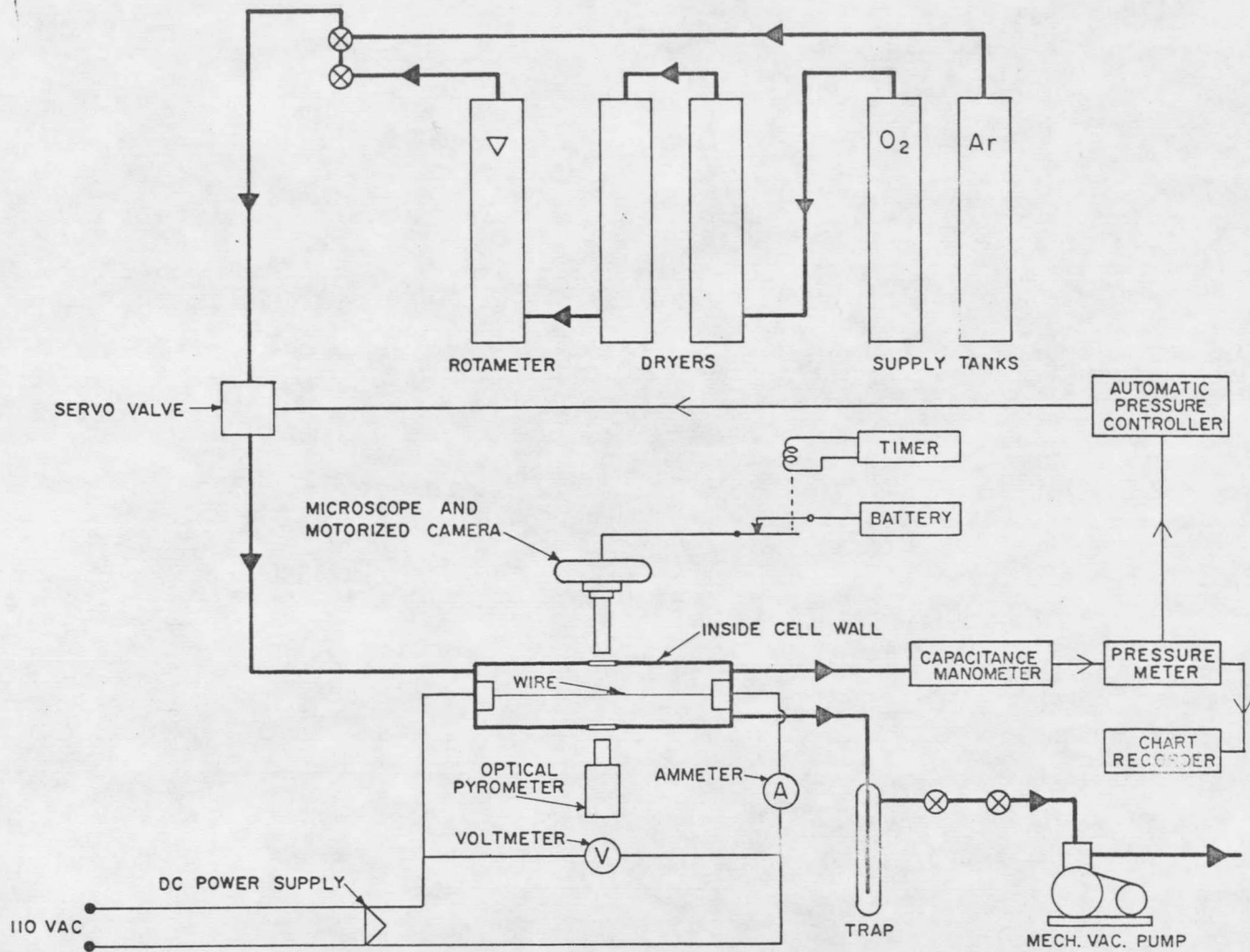


Figure 1. EXPERIMENTAL EQUIPMENT SCHEMATIC

1 inch wide by 3 inches long by 3 inches deep. The electrodes were used to deliver the current to resistance heat the wire. To accommodate the thermal expansion of the wire and thus prevent its bending, a leaf spring type mount was used on one end. This enabled the wire to be loaded tensilely to a pre-determined level such that when the wire was heated to the desired level, the leaf spring would no longer be deflected and thus leave the wire in a near zero stress level.

On the underside of the cell was centered a 4 inch to 1 inch reduction copper plumbing "T", such that the 4 inch diameter main portion of the "T" was centered about a vertical axis through the center of the cell. A 4 inch diameter circular plate was then soldered on the bottom of the "T" to close it off. To the remaining horizontal one inch outlet, was a line leading to the trap and eventually to the mechanical pump. On the opposite side and same elevation as the 1 inch outlet, was located the oxygen gas inlet.

The power to heat the wire was supplied by a Hewlett Packard DC Power Supply, LVR Series, Model 6260B which has the capability of maintaining a constant voltage across or a constant current through whatever it is connected to. In this work the power supply was used so that the voltage drop across the electrodes was constant, which minimized the power change during a run. The current was measured by a DC Ammeter and the voltage was measured by a Honeywell Digital Multimeter, Digitest Model 333R.

Either oxygen or argon was introduced into the cell. The argon was directly connected to the cell through valves, whereas the oxygen was passed through a Drierite Drying Column (to remove any moisture in the oxygen), a #74 Matheson Tube Flowmeter, a manual valve, the servo valve and eventually to the cell. A Granville-Phillips Automatic Pressure Controller No. 0-00-216006 (which included the servo valve), was used in conjunction with a MKS Baratron TM Type 77 Pressure Meter. The MKS unit sensed pressure by means of a MKS Capacitance Manometer 77H-1000. The line tap for the capacitance manometer was located about 2 inches lower and rotated 90 degrees from the inlet for the oxygen, in the wall of the plumbing "T", thus preventing any possible pressure head from the oxygen flow which might cause an incorrect pressure reading.

Gas exiting the cell during a run was passed through a trap designed to condense the oxides present. Liquid nitrogen was used in the trap when the run pressure P_{O_2} , was less than 0.25 atmospheres. When P_{O_2} was greater than 0.25 atmospheres, dry ice was used, since at the temperature of liquid nitrogen and high pressures, P_{O_2} , the boiling point of oxygen is greater than the temperature of the liquid nitrogen. Because of this, if liquid nitrogen were used, the oxygen condensed in the trap. Thus, since the boiling point of the oxygen at the temperature of dry ice and pressure P_{O_2} greater than 0.25 atmospheres is less than the temperature of dry ice, the dry ice was used for P_{O_2} greater than 0.25 atmospheres. From atmospheric, pressure decrease in the cell was achieved by use of a mechan-

ical pump.

All parts of the cell were cooled by cold water circulation through tubes on its surface.

Sight ports were located on opposite sides of the cell at the vertical elevation of a mounted specimen. Shadowgraph quality pyrex windows were used to provide visible access to the cell. These windows were cleaned and polished periodically due to accumulation of oxides on their surfaces. To measure the temperature of the specimen, a Leeds and Northrup 6836-C Optical Pyrometer was used, which was aligned to enable sight through one of the sight port windows. The optical pyrometer was calibrated to take into account the effects of the pyrex windows and thus enable a true temperature reading. This calibration was done as part of the work by Halvorson and Wimber¹³ on the thermal expansion of iridium. Aligned for sight through the other window was a Beseler Topcorn Super D Camera and Electric Motor Drive fitted with a Topcorn Microscope Attachment Model III, which in conjunction with a Lafayette Model 51012T Timer, enabled the taking of a sequence of pictures at a predetermined interval and 20X magnification. For many of the runs, a Lafayette Model 58033, 5-IN-1 Timer was used. The latter timer enabled longer intervals between pictures.

Because of the possibility of power or water loss, the system was fitted with a network of relays which acted as protection devices if water or power supply should be lost during a run.

To perform a run, a 2 7/32 inch section of iridium wire was cut,

cleaned with acetone, ground flat on one end for reference and mounted in the cell. (In the first few runs, rotational pictures were taken of the wire before and after the run. From these it was determined that the eccentricity of the wire was less than 1.2% and thus this procedure was discontinued at that point.) A reference picture was then taken of a drill rod of known diameter (0.0655 cm) in order to provide a reference for the size of the pictures of the iridium wire. An argon atmosphere of 0.92 atmospheres was then introduced into the cell in which the wire was annealed for 30 minutes at 2200 °C. (The anneal was performed upon the use of each new section of wire for the purpose of achieving a grain size and electrical resistivity that would be essentially invariant with time for the runs.) The argon was then removed and the cell filled with oxygen. Then the oxygen was pumped out and a pressure leak test taken to insure a near pure oxygen environment in the cell. The pressure and temperature at which a run was to be made were then set on the equipment. The wire was then oxidized for a short period of time to characteristically texturize its surface. The cycle timer was then activated at its pre-determined cycle interval. A run consisted of ten exposures of the wire, which in total length, corresponded with changing the wire diameter by 0.0015 to 0.0035 cm. (The "0.0015 to 0.0035 cm" range was arrived at because approximately a one mil diameter change was necessary to obtain reasonable data, but if the diameter changed much more than one mil, too much variation in the oxidation rate occurred due to its dependency on the diameter of the wire.)

