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ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR MAY 1973

R. G. Donnelly

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METALS AND CERAMICS DIVISION

ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR MAY 1973

R. G. Donnelly

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AUGUST 1973

OAK RIDGE NATIONAL LABORATORY
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CONTENTS

	Page
IRIDIUM FABRICATION PROGRAM	1
Iridium Sheet Development	1
Preparation of Powder for Melting	1
Electron-Beam Melting and Drop Casting	2
Sheet Rolling	2
Iridium Purification	3
Oxidation of Scrap and Powder	3
Acid Treatment of IrO_2	4
HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES	7
Development of Iridium Alloys	7
Alloy Fabrication	7
Heat Treatment and Recrystallization	7
Tensile Properties	7
Air Oxidation	10
Platinum-Rhodium-Tungsten Alloys	11
Fabrication Development of Platinum-Rhodium-Tungsten Alloys	11
Iridium Transport Studies	11
Thermochemical Effects on Iridium-Tungsten Alloys	13
PHYSICAL AND MECHANICAL METALLURGY OF HEAT-SOURCE CONTAINMENT MATERIALS	15
Effects of Exposure to Oxygen and Carbon on the Physical and Mechanical Properties of Iridium Alloys	15
Multi-Hundred Watt Helium Vent Studies	16
Deposition of Carbon in the Outer Vent	16
Aging of Dissimilar Metal Joints	17
Evaluation of Viking Heat Sources	18

IRIDIUM FABRICATION PROGRAM

Iridium Sheet Development

A. C. Schaffhauser

We are continuing development of procedures for electron-beam melting and fabrication of high-quality iridium sheet for forming into hemispheres. A limited amount of sheet fabricated from the first lot of powder received (Matthey-Bishop) was successfully warm hydroformed at Mound Laboratory. However, sheets fabricated from new lots of powder from the Office of Emergency Preparedness (OEP) have not had the desired grain structure or ductility for good formability. The problem appears to be due to a lower recrystallization temperature and less ductility in the higher purity OEP material. Also, slight variations in trace impurities in different lots of OEP powder result in inconsistent amounts of fibrous grain structure in different sheets. In the meantime, Multi-Hundred Watt Program requirements have shifted and, at the direction of the Space Nuclear Systems Office, we have started fabricating sheet containing 0.25 to 0.30% W to provide improved impact capability. Coincidentally we anticipate that the addition of tungsten will permit us to obtain a consistent fibrous grain structure having good ductility.

Preparation of Powder for Melting – (J. I. Federer)

Approximately 21,600 g of OEP iridium powder (lots 2 and 3, jars 7–18 blend) was used to prepare 144 compacts for electron-beam melting. The powder was washed with distilled water, as previously described,[1] then screened through a 200-mesh screen to separate trash such as bits of sealing wax, brush hairs, and brass wire from previous screening operations. Enough tungsten powder was blended with the iridium powder to make 0.30 wt % W, then compacts weighing about 150 g each were

[1]J. I. Federer, Isotopic Power Materials Development Progress Report for April 1973, ORNL-TM-4263, pp. 1–2.

cold pressed at 50 tsi in a 1 × 1.8 in. die. The compacts were presintered at 1000°C for 1 hr in hydrogen, then vacuum sintered at 1500°C for 4 hr. Compacts numbered 19 through 162 were prepared in this manner.

Electron-Beam Melting and Drop Casting – (R. E. McDonald)

Thirty Ir–0.30% W ingots were melted from the new 1200-oz lot of OEP powder during the month using the procedures described previously.[2] The first three ingots in this series (WC-1, -2, -3) were each melted with approximately 900 g of sintered compacts and 180 g of skull from ingot WB-2 to minimize the amount of additions from previous lots of powder in these ingots. All subsequent ingots were melted with 450 g of sintered compacts and 600 g of heads and skulls from ingots previously melted in this series.

Sheet Rolling – (R. W. Knight)

Fabrication studies for manufacturing sheet from Ir–0.25% W and Ir–0.30% W have continued. Flowsheets have been written for ingot preparation and rolling.

Ingot preparation includes acid cleaning, acetone cleaning in an ultrasonic bath, shrink hole welding, x ray, homogenizing and conditioning the ingots by grinding the top and bottom surface of what will be the finished plate.

Ingot rolling is done in three steps; each step requires resizing the molybdenum frame and wrapping in a new molybdenum cover sheet. The first rolling schedule consists of four passes at 25% each, two passes longitudinal and two passes transverse. After this schedule the ingots are recrystallized at 1200°C for 1 hr. The second rolling schedule requires three passes – two at 25% and one at 17% reduction – all cross rolled. The first pass is at 1100°C and the last two at 1000°C. This gives an ingot width of 2 7/8 in. The final rolling schedule is at 10% reduction per pass with all passes

[2] R. E. McDonald, ibid., pp. 3–4.

in the longitudinal direction. Approximately 50% of the passes are at 1000°C and the final 50% at 950°C, yielding a sheet approximately 11 × 3 1/4 × 0.031 in. All sheets are stress relieved to flatten the sheets for Eloxing and grinding.

The hardness is measured after each rolling schedule and after recrystallization as a measure of repeatability of the rolling of the sheet or to identify any that differ.

Sheets EBP-17 and -19, which had additions of 300 to 600 ppm W, were fabricated using parameters previously reported.[3] These finished sheets showed a better wrought structure than EBP-16, -17, -20, -21, which were reported previously.

During this month the following sheets were rolled, using the parameters described above: WA-3, -4, -6 (0.25% W) for hydroforming studies at Mound Laboratory; WA-5 for chemistry standards; WB-1, -2, -3 (0.30% W) for hydroforming studies at Mound Laboratory; WC-1 through -26 for hydroforming blanks. Sheets WC-1 through -12 have been ground and sent to Mound Laboratory.

Early in the WC series, sheets were cold rolled up to about 0.0015 in. to flatten for machining. The varying amount of cold work in these sheets could account for the flatter fibrous microstructures observed in some sheets. Sheets presently are stress relieved between platens to provide a more uniform microstructure.

Iridium Purification

J. I. Federer

Oxidation of Scrap and Powder

During the past month seven oxidation tubes operated for periods ranging from 60 to 100% of full time. The IrO_2 production rate and status of all tubes are shown in Table 1. The average production rate for all tubes was 8.1 g IrO_2/tube compared to an average of 11.1 g IrO_2/tube for the previous month (April). The total amount of IrO_2

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[3] G. A. Reimann and R. W. Knight, ibid., pp. 4-6.

Table 1. IrO_2 Production During May 1973

Oxidation Tube[a]	IrO_2 Produced[b] (g)	Days Operated	Average IrO_2 Production Rate (g/day)	Status
1	0	0	0	Shut down for recharging
2	151	31	4.9	Operating
3	170	24	7.1	Shut down temporarily, then recharged
	356	31	11.5	Operating
5	120	18	6.7	Shut down for recharging
6	156	31	5.0	Operating
7	0	0	0	Shut down for recharging
8	0	0	0	Shut down for recharging
9	378	31	12.2	Operating
10	0	0	0	Shut down for recharging
11	290	31	9.4	Operating
Total	1621			

[a]Tubes 2, 3, 9, and 11 contained Engelhard scrap. During the month tube 3 was recharged with compacted Goldsmith powder. Tube 4 contained compacted Goldsmith powder. Tube 6 contained hemisphere scrap.

[b]All tubes operated at 1100°C. The oxygen flow rate was 2000 cm^3/min except 1000 cm^3/min from Friday (4:00 p.m.) until Monday (9:00 a.m.).

produced was only 1621 g compared to 3591 g during the previous month. This decline in production rate is due to a decrease in the amount of material available for purification.

Acid Treatment of IrO_2

Approximately 5000 g of IrO_2 prepared from Engelhard scrap was crushed as needed to −40 mesh, then exposed to the following acid solutions: 50% HF–50% H_2O, concd HCl, aqua regia (1 part concd HNO_3–3 parts concd HCl), concd HNO_3, and concd H_2SO_4. The HF solution was used at room temperature, the aqua regia at 40 to 50°C, and the others at about 90°C. The powder, in beakers containing about 500 g each, was exposed first to the HF solution for at least 48 hr to dissolve

silica glass contamination from the oxidation tubes. The time of exposure to the other acids was 16 to 20 hr. After exposure to each of the first four acids, the powder was washed with distilled water five times. Washing was accomplished by vigorous mixing to form a slurry, which was then allowed to settle before decanting. After the last acid treatment the powder was washed eight to ten times, at which point settling became very sluggish. The powder was then evaporated to dryness. If acceptable chemical analysis is obtained, the IrO_2 will be reduced to metal with hydrogen at 800°C.

HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES

Development of Iridium Alloys

C. T. Liu

Alloy Fabrication

The fabrication of three 150-g iridium alloy ingots containing up to 4 wt % Re, Ru, and W has been delayed four weeks by the production of iridium sheet for the Iridium Fabrication Program.

Heat Treatment and Recrystallization

To determine the recrystallization temperature and softening behavior, Ir–1% Ru, Ir–1% Nb, and Ir–0.93% Hf alloy sheet, fabricated at 1100°C, were vacuum annealed 1 hr between 900 and 1600°C. These are being prepared for metallographic examination and microhardness measurements.

Tensile Properties

Tensile specimens with a gage section of 1/2 × 1/8 in. were stamped from the sheets at room temperature, annealed 1 hr at 1500°C, and tested at temperatures up to 1316°C (2400°F) in vacuum. The newly obtained tensile data from Ir–0.5% Nb, Ir–1% Nb, and Ir–0.93% Hf are presented in Table 2 together with those for pure iridium and binary iridium alloys reported previously. The effects of Ru, Re, W, Nb, and Hf on the tensile properties of annealed iridium are as follows:

1. All alloys have a lower room-temperature ductility than that of pure iridium (12.6%).

2. The lower room-temperature ductility of the iridium alloys is partially due to their higher yield strength. For instance, Ir–0.93% Hf alloy has only 3.2% elongation at room temperature; however, its yield strength is 43.7 ksi, which is higher than iridium by 500%.

3. A plot of the ductility as a function of temperature shows that alloying with up to 2% W improves the ductility of iridium in the intermediate temperature range (200 to 800°C).

Table 2. Tensile Properties of Iridium and Iridium Alloy Sheet[a]

Alloy (wt %)	Strength, ksi Yield	Strength, ksi Tensile	Elongation (%)
		Room Temperature	
Ir[b]	8.8	63.0	12.6
Ir–0.5% Ru	11.0	24.0	5.2
Ir–1% Ru	13.5	34.0	7.0
Ir–1% Re	25.5	48.0	6.6
Ir–1.98% Re	26.0	67.6	8.9
Ir–1% W	29.1	56.0	6.8
Ir–1.92% W	36.7	70.3	8.3
Ir–0.5% Nb	25.0	56.5	7.2
Ir–1% Nb	39.6	60.0	5.1
Ir–0.93% Hf	43.7	50.9	3.2
		500°C	
Ir[b]	14.3	43.0	15.0
Ir–0.5% Ru	13.3	38.8	11.1
Ir–1% Ru	14.5	35.0	10.0
Ir–1.98% Re	26.2	62.5	13.7
Ir–1% W	26.0	63.0	13.8
Ir–1.92% W	28.0	93.3	21.5
Ir–0.5% Nb	20.5	70.9	14.7
		760°C	
Ir–0.5% Ru	9.0	42.0	17.0
Ir–1% Ru	15.4	47.0	23.0
Ir–1% Re	22.0	50.0	20.0
Ir–1.98% Re	19.3	67.3	28.0
Ir–1% W	23.6	58.0	24.4
Ir–1.92% W	22.2	84.2	30.0
Ir–0.5% Nb	16.5	68.6	21.8
Ir–1% Nb	20.6	73.7	17.0
Ir–0.93% Hf	30.7	66.5	10.6
		1093°C	
Ir[b]	10.4	35.1	49.4
Ir–0.5% Ru	8.0	33.5	50.3
Ir–1% Ru	12.5	31.4	35.5
Ir–1% Re	16.5	37.0	43.0
Ir–1.98% Re	20.0	42.7	51.1
Ir–1% W	21.0	40.0	41.8
Ir–1.92% W	22.2	60.5	31.2
Ir–0.5% Nb	13.7	49.6	31.0
Ir–1% Nb	17.8	58.9	24.0
		1316°C	
Ir[b]	5.5	26.3	50.2
Ir–0.5% Ru	8.5	22.5	42.6
Ir–1% Ru	8.0	23.0	45.4
Ir–1% Re	15.2	26.7	43.8
Ir–1.98% Re	16.0	31.3	47.0
Ir–1% W	16.0	29.5	43.5
Ir–1.92% W	17.0	45.2	36.0
Ir–0.5% Nb	13.3	37.5	32.3
Ir–1% Nb	14.8	46.8	30.3
Ir–0.93% Hf	23.0	70.0	37.4

[a]Tested in vacuum at a crosshead speed of 0.5 to 0.1 in./min. All specimens annealed 1 hr at 1500°C in vacuum prior to testing.

[b]Sheet EB-5.

4. Hafnium is the most effective addition that improves the strength of iridium. Iridium–0.93% Hf has a tensile strength of 70.0 ksi and a yield strength of 23.0 ksi at 1316°C, which are higher than unalloyed iridium by 300 to 400%. Iridium–0.93% Hf alloy is stronger than all the existing candidate alloys for space isotopic heat sources.

5. All the iridium alloys have ductilities more than 30% at 1316°C.

6. The strengthening effect of the alloying element decreases in the order – Hf, Nb, W, Re, and Ru. Up to 1%, ruthenium has no strengthening effect on iridium.

The toughness of iridium and iridium alloys at 1316°C was calculated and is compared with other candidate alloys in Table 3. The toughness of Ir–0.93% Hf is the best and is about 200% higher than unalloyed iridium. Iridium–1.92% W and Ir–1.98% Re rank next and are comparable to T-111 and Pt–30% Rh–10% W.

Table 3. Comparison of Toughness[a] of Developmental Iridium-Base Alloys with Other Candidate Alloys[b] at 1316°C (2400°F)

Composition (wt %)	Toughness (in.-lb/in.^{3})
Ir[c]	8,000
Ir–0.5% Ru	6,600
Ir–1% Ru	7,000
Ir–1% Re	8,800
Ir–1.98% Re	11,100
Ir–1% W	9,900
Ir–1.92% W	11,200
Ir–0.5% Nb	8,200
Ir–1% Nb	9,300
Ir–0.93% Hf	17,400
Pt–30% Rh	2,100
Pt–30% Rh–8% W	9,200
Pt–30% Rh–10% W	10,900
TZM	6,100
T-111	11,000

[a]Defined as fracture strain × (tensile strength + yield strength)/2.

[b]H. Inouye, C. T. Liu, and R. G. Donnelly, New Platinum-Rhodium-Tungsten Alloys for Space Isotopic Heat Sources, ORNL-4813 (1972).

[c]Sheet EB-5.

Air Oxidation

To continue the study of the air oxidation properties of iridium alloys, Ir–0.93% Hf, Ir–1% Ru, and Ir–1% Nb, sheet specimens 1 × 0.5 × 0.025 in. were annealed 1 hr at 1500°C and oxidized in air at a flow rate of 100 liters/hr at 770, 870, and 1000°C. The oxidation at 1000°C was terminated after 330 hr exposure because the oxide scale and material on the edge and surface of the specimens flaked markedly in the form of powders. At present, the oxidation runs at 700 and 870°C have logged 770 hr. The results are presented in Table 4 and discussed below.

1. Oxidation at 770°C. Light-brown oxide spots gradually appeared on the specimen surface at 770°C. All the specimens showed a small initial weight loss within 100 hr exposure, followed by a small weight increase. Since the weight change (at a rate of about 1.7×10^{-8} g cm^{-2} hr^{-1}) for all the alloys is so small, an accurate oxidation rate could not be measured.

Table 4. Oxidation of Iridium Alloys in Flowing Air[a] at 770, 870, and 1000°C

Alloy (wt %)	Oxidation Rate, g cm^{-2} hr^{-1}, at		Total Weight Change, g/cm^3, After Exposure for	
	330 hr	770 hr	330 hr	770 hr
		770°C		
Ir–1% Ru	b	b	b	-7.1×10^{-6}
Ir–1% Nb	b	b	b	$+1.4 \times 10^{-5}$
Ir–0.93% Hf	b	b	b	$+5.8 \times 10^{-6}$
		870°C		
Ir–1% Ru	-8.6×10^{-7}	-8.6×10^{-7}	b	-7.7×10^{-4}
Ir–1% Nb	$+7.2 \times 10^{-8}$	$+7.2 \times 10^{-8}$	b	$+6.5 \times 10^{-5}$
Ir–0.93% Hf	-7.1×10^{-7}	$+7.1 \times 10^{-7}$	b	-5.4×10^{-4}
		1000°C		
Ir–1% Ru	-4.7×10^{-4}	c	-1.6×10^{-1}	c
Ir–1% Nb	-4.6×10^{-4}	c	-1.5×10^{-1}	c
Ir–0.93% Hf	-3.3×10^{-4}	c	-1.1×10^{-1}	c

[a]Flow rate of 100 liters/hr.

[b]Weight changes too small.

[c]No test.

2. Oxidation at 870°C. A dark-brown oxide layer is observed on the specimen surfaces at 870°C. The iridium-ruthenium and iridium-hafnium alloys show a continuous weight loss at a rate of 8.6 and 7.1×10^{-7} g cm^{-2} hr^{-1}, respectively, whereas iridium-niobium shows a weight gain at a rate of 7.2×10^{-8} g cm^{-2} hr^{-1}. The rate of weight loss in iridium-ruthenium and iridium-hafnium is about equal to pure iridium (1.1×10^{-6} g cm^{-2} hr^{-1}). However, the kinetics of the weight change in the iridium-niobium alloy is lower than pure iridium by a factor of 15.

3. Oxidation at 1000°C. A dark-brown oxide which formed on the specimens was not adherent and began to flake off after a short exposure at 1000°C. The weight decrease at 1000°C is linear with time for the three alloys. The oxidation rate of Ir–1% Ru and Ir–1% Nb is about equal and a little higher than Ir–0.93% Hf alloy and pure iridium (3.2×10^{-4} g cm^{-2} hr^{-1}).

Platinum-Rhodium-Tungsten Alloys

H. Inouye

Fabrication Development of Platinum-Rhodium-Tungsten Alloys

Electron-beam purification of platinum and rhodium melting stock has been initiated. High-purity tungsten powder compacts have been pressed and sintered preparatory to the melting of a W–50% Pt master alloy. A Pt-3008 (Pt–30% Rh–8% W) billet which was induction melted as a 2400 g ingot (4 × 4 × 0.5 in.) by Matthey-Bishop is ready to be rolled to sheet. Both melting and rolling requests have been delayed about three weeks because of the Iridium Sheet Development Program.

Iridium Transport Studies

H. Inouye

Vent plugging of PuO_2-fueled iridium spheres is attributed, in part, to the formation of volatile iridium oxides and its dissociation

in a temperature gradient back to iridium. The source of the oxygen is thought to be the fuel, the iridium, and/or other impurities. To provide information that may be applicable to the above problem, four methods were evaluated as to their effectiveness in removing oxygen and metallic impurities from the surface of fabricated iridium at temperatures below its recrystallization temperature. Table 5 lists the results of spark-source spectrographic analyses of the surface and an oxygen analysis of total samples. It is obvious from the results that this particular piece of iridium was severely contaminated with oxygen and several other impurities which were removed with various degrees of success by all methods. Most notable was the lack of any improvement in reducing the concentration of iron and aluminum. From the standpoint of oxygen removal, these results indicate that electro-cleaning with KCN was most effective.

Table 5. Analyses[a] of Engelhard Scrap Iridium Surfaces Cleaned by Various Methods

Impurity	As-Received	Cleaning Method[b]			
		A	B	C	D
Al	> 1%	> 1%	> 1%	> 1%	> 1%
Ca	2000	200	400	100	600
Co	60	6	2	6	60
Cr	600	600	200	200	60
Fe	> 1%	> 1%	> 1%	> 1%	1%
Mg	800	3	3	3	30
Mn	60	20	6	60	60
Mo	200	60	6	60	60
Ni	500	170	170	500	170
O	73	45	10	34	23
Pt	3000	3000	300	300	3000
Rh	> 1000	> 1000	300	300	1000
S	250	80	25	3	800
Si	20	2	6	6	20
Th	250	80	80	8	30
Ti	1500	1500	30	100	100
W	200	200	200	200	600

[a]Analyses in parts per million except as noted.

[b]Method A — Soak 16 hr in HF at room temperature plus 4 hr in hot aqua regia. Method B — Electro-clean in 2N-KCN 50 min at 5 V ac. Method C — Method A plus 2 hr in hydrogen at 900°C. Method D — Heat in Cl_2 for 2 hr at 850°C, cool in argon.

Table 6 gives the results of measurements on the removal rate of iridium when electro-cleaned in KCN at 5 V ac. These data show that precise control can be maintained on the amount of iridium removed by regulating the cleaning time. At 5 V ac the removal rate is calculated to be 1.03×10^{-5} in./min or 0.0005 in. from the surface of the iridium sample. Since the samples were treated under these conditions for 50 min (Table 5), these results therefore show that the depth of iron, aluminum, and tungsten contamination in this material is greater than 0.0005 in. The other listed elements appear to be surface impurities.

Table 6. Dissolution of Iridium (Heat EB-10A) During Electro-cleaning in 2N-KCN at 5 V ac[a]

Cleaning Time (min)	Sample Weight (g)	Dissolution Rate[b] ($g\ cm^{-2}\ min^{-1}$) ($\times 10^{-4}$)
0	1.6750	
5	1.6673	5.70
10	1.6590	5.93
15	1.6511	5.90
25	1.6350	5.92
35	1.6194	5.88

[a]0.0221-in.-thick sheet; area = 2.7 cm^2.

[b]Average = 5.87×10^{-4} g cm^{-2} min^{-1}.

= 2.6×10^{-5} cm/min.

= 1.03×10^{-5} in./min.

Thermomechanical Effects on Iridium-Tungsten Alloys

C. T. Liu

The effects of thermomechanical treatments and small amounts of tungsten (at the levels of 0.1, 0.3, and 0.5 wt % W) on the recrystallization, structure, and mechanical properties of iridium are being determined. The Ir-0.1% W and Ir-0.5% W alloy compacts have been

prepared by sintering of mixed powders at 1500°C; they are now ready for melting and casting into 150-g rectangular ingots. Iridium alloy sheets containing 0.25 and 0.30 wt % W were obtained from the Iridium Fabrication Program.

PHYSICAL AND MECHANICAL METALLURGY OF HEAT-SOURCE CONTAINMENT MATERIALS

Effects of Exposure to Oxygen and Carbon on the Physical and Mechanical Properties of Iridium Alloys

C. T. Liu

Tensile specimens of Ir–1.92% W and Ir–1.98% Re heat treated 1 hr at 1500°C were contacted with ATJ graphite on one side and exposed to an oxygen pressure of 1×10^{-5} torr at 1300°C in a mullite tube furnace. Both sides of the specimens are bright and show no indication of reaction with graphite and oxygen after a 1000-hr exposure. Table 7 gives the weight change in the exposed specimens. The specimens generally showed a small weight loss (except for one Ir–1.98% Re specimen) at an average rate of –14 and –15 ppm for iridium-tungsten and iridium-rhenium alloys, respectively. It is

Table 7. Weight Change of Iridium Alloy Sheet Specimens Contacted with Graphite on One Side and Exposed to Oxygen at 1×10^{-5} torr for 1000 hr at 1300°C

Alloy (wt %)	Weight Change, ppm, in Specimens					
	A	B	C	D	E	F
Ir–1.92% W	–14	–1	–14	–19	–21	–13
Ir–1.98% Re	–16	+3	–21	–18	–18	–20

then expected that any change in interstitial content due to exposure is very limited. The results of tensile tests of the exposed specimens are presented in Table 8 together with those for unexposed specimens. The exposure to oxygen and graphite at 1300°C did not impair the ductility of iridium alloys at all test temperatures. The tensile strength was not sensitive to oxygen and graphite exposure; however, the yield strength of the exposed specimens is lower than the unexposed ones. Note that lowering in yield strength is more pronounced in the iridium-rhenium alloy. These data indicate that Ir–1.92% W and Ir–1.98% Re alloys, as well as pure iridium, are compatible with the "simulated heat source" environment at 1300°C.

Table 8. Tensile Properties[a] of Iridium Alloy Sheet Specimens[b] Contacted with ATJ Graphite on One Side and Exposed to Oxygen at 1×10^{-5} torr at 1300°C

Alloy (wt %)	Exposure Time (hr)	Strength, ksi Yield	Strength, ksi Tensile	Elongation (%)
		Room Temperature		
Ir–1.92% W	0[c]	36.7	70.3	8.3
	1000	33.7	68.2	7.9
Ir–1.98% Re	0[c]	26.0	67.6	8.9
	1000	21.4	56.1	8.4
		760°C		
Ir–1.92% W	0[c]	22.2	84.2	30.0
	1000	19.6	85.0	28.3
Ir–1.98% Re	0[c]	19.3	67.3	28.0
	1000	13.6	67.2	28.2
		1093°C		
Ir–1.92% W	0[c]	22.2	60.5	31.2
	1000	17.8	60.8	41.4
Ir–1.98% Re	0[c]	20.0	42.7	51.0
	1000	12.0	41.0	44.6
		1316°C		
Ir–1.92% W	0[c]	17.0	45.2	36.0
	1000	16.0	47.1	37.0
Ir–1.98% Re	0[c]	16.0	31.3	47.0
	1000	8.0	28.7	55.7

[a]Tested in vacuum at a crosshead speed of 0.05 to 0.1 in./min.

[b]All specimens annealed 1 hr at 1500°C prior to exposure to graphite and oxygen.

[c]Unexposed specimens vacuum annealed 1 hr at 1500°C prior to tensile testing.

Multi-Hundred Watt Helium Vent Studies

H. Inouye

Deposition of Carbon in the Outer Vent

The experiment concerned with the transport of carbon in a temperature gradient via the reaction $2\ CO \rightarrow CO_2 + C$ was terminated

after 2296 hr due to the development of a large leak in the quartz bulb. A summary of the helium flow rates through a quartz capillary to 2111 hr is listed in Table 9. These data show only about a 2% reduction in the conductance for the first 950 hr. Between 950 and 2111 hr the conductance of the vent decreased by an additional 14% (a factor of about 7 higher). A total reduction of about 16% was measured during the test. A postmortem will be conducted to determine the location of the carbon deposit.

Table 9. Helium Flow Rate Through a Quartz Vent[a]

Exposure Time (hr)	P_{CO} in Bulb (atm)	$t_2 - t_1$[b] (sec)	Percent of Original Flow Rate
0	0	52.92	100.00
459	0.346	54.05	97.90
600	0.293	54.42	97.24
787	0.266	54.00	98.00
950	0.532	54.00	98.00
1112	0.532	55.42	95.48
1275	0.532	56.35	93.91
1436	0.532	57.83	91.35
1623	0.532	59.10	89.43
1759	0.532	59.82	88.46
1924	0.532	62.42	84.78
2111	0.532	63.30	83.60

[a]Vent dimensions: 0.125 in. ID × 2 in. long plus a 0.020-in.-diam Pt–10% Rh wire in a capillary, 0.035 in. ID × 18 in. long.

[b]Time to exhaust helium from a 12.6 cm^3 bulb from 650 to 0.020 torr.

Aging of Dissimilar Metal Joints

The aging behavior of an Ir/Pt–20% Rh butt weld is being studied to determine whether the expected two-phase structure will become brittle. The hardness gradients across the weld for aging times to 2000 hr at 538°C are listed in Table 10. The hardness of the aged welds on the platinum side continue to be higher than in the as-welded condition and shows no tendency to decrease with time. However, when

Table 10. Hardness[a] Gradient Across an Ir/Pt–20% Rh Weld

Distance from Iridium Fusion Line (mils)	As-Welded (DPH)	Aged at 538°C for 1000 hr (DPH)	Aged at 538°C for 2000 hr (DPH)	Aged 24 hr at 850 and 538°C for 1000 hr (DPH)	Aged 24 hr at 850 and 538°C for 2000 hr (DPH)
Iridium base metal	380	355	325	330	300
0 (fusion line)	300	300	305	295	295
2	305	302	298	284	245
4	315	297	298	284	250
6	325	293	297	288	257
8	327	292	300	293	257
10	333	290	315	297	258
12	321	288	325	300	260
14	305	290	310	302	262
16	290	298	300	300	257
18	275	300	300	295	247
20	260	297	302	292	241
22	245	295	310	288	240
24	237	293	307	281	235
26	233	289	290	268	233
28	183	260	235	250	235
30 (fusion line)	134	130	175	157	175
Pt–20% Rh base metal	132	131	141	120	122

[a]0.030-in.-thick sheet, annealed 1 hr at 1100°C, electron-beam welded, 500-g load.

the weld is given a double heat treatment of 24 hr at 850°C then aged at 538°C, the hardness after 2000 hr is significantly lower than that at any other condition.

Evaluation of Viking Heat Sources

H. Inouye

The effect of a design change in the Viking heat source that results in an increase in temperature of the T-111 strength member from 825 to 870°C was evaluated. The temperature increment changed the estimated contamination rate from 23.4 to 29.2 ppm O/1000 hr.

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