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ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR DECEMBER 1973

R. G. Donnelly

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ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR DECEMBER 1973

R. G. Donnelly

MAY 1974

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IRIDIUM FABRICATION PROGRAM

A. C. Schaffhauser

Fabrication of Material for PICS

We are continuing production of Ir–0.3% W forming blanks for the postimpact containment shells (PICS) of the MHW Isotope Heat Source. Fabrication of blanks from the WC batch of powder is completed and fabrication of blanks from recycled sheet scrap is underway. We have also melted and fabricated three ingots from the WE-batch of powder.

Preparation of Compacts for Melting — (J. I. Federer)

Approximately 11,400 g of OEP iridium powder from the WE batch was compacted for electron beam melting. The compacts were prepared by blending powder from 12 jars of higher purity powder.[1] The usual procedures of water washing of the iridium powder, blending of iridium with 0.3 wt % tungsten powder, pressing, and presintering were used. The tungsten content of the blended powders was analyzed by the atomic absorption technique, which has an accuracy of about ±10%. The analyzed tungsten contents ranged between 0.32 and 0.35 wt %.

Blank Production — (R. W. Knight)

A summary of data on blanks manufactured during the month is given in Table 1. A total of 36 WC-series production blanks, including four having nonconformance reports, were shipped to MRC. In addition 10 WCR blanks from recycled sheet were shipped for forming evaluation.

Fabrication Development for MHW Outer Clad

We are developing a backup capability for fabrication of the Multi-Hundred Watt heat-source iridium outer clad. Our objective is to develop

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[1]J. I. Federer, *Isotopic Power Materials Development Progr. Rep. for Nov. 1973*, ORNL-TM-4513, p. 2.

Table 1. Disposition and Characteristics of Ir–0.3% W Blanks

Ingot Number	Number Manufactured	Rejections			Number Shipped	Average Hardness (DPH)	Tungsten Content (ppm)
		Ultrasonic	Penetrant	Surface			
WC-67	4	0	0	0	4	540	4000
WC-68	4	0	1	0	3	538	3500
WC-69	4	0	1	0	3	521	3500
WC-70	4	0	0	1	3	530	4000
WC-71	4	2	1[a]	0	2	539	2500
WC-72	4	1[a]	0	0	4	532	2200
WC-73[b]							
WC-74	4	1	0	0	3	520	3200
WC-75[b]							
WC-76	4	1[a]	0	0	4	517	2500
WC-77	4	0	0	0	4	526	2500
WC-78	4	1	0	0	3	530	3100
WC-79	4	1,1[a]	0	0	3	537	3800
WCR-80	4	0	1[a]	0	0[c]	527	3800
WCR-81	4	2	0	1	0	464	3400
WCR-82	4	1	0	0	3		3600
WCR-83	3	0	0	0	3		2900
WCR-84	4	0	0	0	4		3100

[a]Nonconformance.

[b]Used for end cap stock.

[c]Held for further evaluation

[d]Rejected for partial recrystallized structure due to furnace over temperature.

alternate fabrication techniques to those currently being used commercially in order to obtain a more reproducible product.

Forming of Iridium End Caps — (R. W. Knight and D. S. Parker[2])

Our original approach to improved fabrication of end caps was to hot form single piece end caps in graphite dies at 1100°C. This technique was successfully demonstrated using 0.030-in. thick sheet rolled from pure iridium ingots on hand. The processing steps have been described previously. Tolerances of ±0.010-in. were obtained on the critical diameters and 0.040 to 0.060-in. radii were achieved in the dish and weld lip areas. However, later design specifications required tighter tolerances on the diameters and sharper radii in dish and weld lip areas. These tighter tolerances require a final warm coining operation in metal dies. A minimum coining temperature of 800°C is desirable. However, due to the unavailability of low expansion high temperature die material for this temperature, coining was attempted in cast iron dies at 600°C. Lid PL-2 was successfully warm coined using this technique. However lids PL-3, -4, and -6 contained surface cracks after this operation. Attempts to remove these cracks by surface conditioning were not successful.

In an attempt to develop a more reproducible process, the following changes are being made:

1. Ir—0.3% W will be used for end caps to obtain better grain size control and fabricability.

2. Sheet for forming blanks will be final rolled bare (no cover sheets) to obtain a smooth surface and better thickness control.

3. A low expansion, high temperature die material, Inconel 903, has been ordered for final coining at 800°C.

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[2]Union Carbide Corporation, Nuclear Division, Y-12 Plant, Oak Ridge, Tennessee.

HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES

H. Inouye

Development of Iridium-Base Alloys

C. T. Liu and H. Inouye

Tensile Properties

We had difficulties in blanking good tensile specimens of Ir–8% Re and Ir–10% Ru alloys from the hot fabricated sheets. This problem may be solved by properly annealing the sheets prior to punching the specimens.

Air Oxidation

A study of the air oxidation of Ir–0.65% Hf, Ir–10% Ru, and Ir–8% Re alloys was initiated. The sheet specimens heat treated 1 hr at 1500°C are being oxidized at an air flow rate of 100 liters/hr at 770, 870, and 1000°C. At present, the oxidation runs have logged 200 hr.

Scaleup of Iridium-Base Alloys

C. T. Liu and H. Inouye

Tensile tests were performed to characterize the mechanical properties of Ir–1.92% W sheets fabricated from 400-g (3/4 × 3/4 × 2 1/8 in.) rectangular ingots. Tensile specimens were blanked from the Ir-38 sheet stock and recrystallized 1 hr at 1500°C, then tested at temperatures up to 1370°C in vacuum. The tensile results are presented in Table 2. Compared with the tensile properties[3] of the Ir–1.92% W alloy sheet fabricated from 150-g (0.3 × 1 × 2 in.) ingot, both the yield and tensile strengths of Ir-38 sheet are a little lower; however, the elongation is higher, especially above 760°C. In terms of toughness, defined as [fracture strain × (yield + tensile)/2], Ir-38 sheet is slightly higher (13,900 in.-lb/in.^{3}) than the sheet fabricated from the 150 g ingot.

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[3]R. G. Donnelly, *Isotopic Power Materials Development Progress Report for April 1973*, ORNL-TM-4263.

Table 2. Tensile Properties[a] of Ir–1.92% W Alloy Sheet[b]

Test Temperature (°C)	Strength, ksi		Elongation (%)
	Yield	Tensile	
Room	32.8[c]	64.0[c]	8.4[c]
760	21.2[d]	79.8[d]	29.1[d]
1093	20.4[d]	55.2[d]	40.2[d]
1370	15.3[d]	38.0[d]	52.3[d]

[a]Tested in vacuum at a crosshead speed of 0.05 to 0.2 in./min.

[b]All sheet specimens annealed 1 hr at 1500°C prior to testing

[c]Data obtained from the specimen parallel to the rolling direction.

[d]Data obtained from the specimens perpendicular to the rolling direction.

Three Ir–1.98% Re ingots, 3/4 × 3/4 × 2 1/8 in., were prepared from electron-beam melting of 3300 g alloy coupons obtained from the iridium purification program. The drop-cast ingots were cleaned in an ultrasonic bath and pickled in an acid solution. The shrink holes on the cropped end of the ingots were filled by electron-beam welding.

Ruthenium Characterization and Fabrication Development

H. Inouye

Ruthenium has a high melting point (2310°C), a moderate density (12.45 g/cm^3), and is considerably less expensive (about $2/g) than iridium, platinum, or rhodium. Reports indicate that ruthenium exhibits air oxidation rates lower than iridium below 1000°C (ref. 4) and is nonreactive to carbon to 1840°C (ref. 5). The tensile strength of ruthenium is

[4]W. L. Phillips, *Trans. ASM* 57, 33-37 (1964).

[5]J. F. Collins and N. McConnaughey, *Investigations on Workability of Unalloyed Ruthenium*, GESP-720 (Dec. 30, 1971).

comparable to or slightly lower than iridium.[6] These characteristics indicate that ruthenium and its alloys could be a promising cladding material for isotopic fuels. Investigations to date, however, show that ruthenium tends to be brittle and consequently promises to be a most difficult metal to fabricate.[5,6,7] This task strives to verify the properties of unalloyed ruthenium and develop a process for producing a high quality sheet.

Ruthenium powder from Engelhard (lot Ru-21-23), with the analysis shown in Table 3, was pressed at 15 tsi into compacts about 1.8 × 1.0 × 0.2 in. Four compacts of about 40 g each had an average density of 57.7% of theoretical after sintering for 2 hr in hydrogen at 1000°C. The average density after a second sinter in vacuum for 4 hr at 1500°C was 86.1% of theoretical. The compacts will ultimately be electron-beam melted into 80 g buttons.

Platinum-Rhodium-Tungsten Alloys

H. Inouye

Fabrication Development

Arc-melted and drop-cast ingots of Pt-3008 and Pt-2608, using the rhodium from lot 96876, cracked severely on hot rolling, and sheet produced from such an ingot has poor high-temperature ductility.[8] To determine whether the abnormally poor properties can be eliminated by electron-beam melting, a 900-g charge will be electron-beam melted and drop cast using scrap from the cracked ingots. A second ingot using scrap from normal heats will also be electron-beam melted to determine whether the ductility minimum around 1000 to 1100°C can be eliminated.

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[6]R. W. Douglass et al., *High Temperature and Alloying Behavior of Refractory Pt-Group Metals*, BMI NP 10939 (August 1961).

[7]D. W. Rhys, *J. Less Common Metals* 1, 269-291 (1959).

[8]R. G. Donnelly, *Isotopic Power Materials Development Progress Report for October 1973*, ORNL-TM-4445.

Table 3. Spectrographic Analysis of Engelhard Ruthenium Powder, Lot Ru-21-23

Element	Weight (ppm)	Element	Weight (ppm)
Al	8	Nb	10
B	0.1	Ni	2
Ba	0.1	P	0.1
Ca	3	Si	2
Co	0.2	Ta	5
Cr	2	Ti	0.6
Cu	1	Zn	0.2
Fe	5	Zr	0.2
K	0.3	Cl	5
Li	0.1	S	1
Mg	0.8		
Mn	2		

Fabrication of Compatibility Components — (H. Inouye)

Rod products 9/16 and 3/16 in. in diameter (heat Pt-5A) and sheet stock 0.050 and 0.035 in. thick (heat Pt-5B) were fabricated from two arc-melted ingots. The above items were delivered to J. R. DiStefano on December 14, 1973, for the fabrication of capsule assemblies for the Curium Program. A third ingot (heat Pt-5C) has been arc melted as a replacement for heat Pt-4X which was determined to be unsuitable for tensile specimen due to its poor ductility.[9]

[9]R. G. Donnelly, *Isotopic Power Materials Development Progr. Rep. for Nov. 1973*, ORNL-TM-4513.

PHYSICAL AND MECHANICAL METALLURGY OF HEAT-SOURCE CONTAINMENT MATERIALS

H. Inouye

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

C. T. Liu

To characterize the compatibility between iridium-base alloys and the simulated heat-source environment, tensile specimens of Ir—3.84% W and Ir—2% Ru contacted with ATJ graphite on one side were exposed to an oxygen pressure of 1×10^{-5} torr at 1300°C. Both sides of the specimens were bright and showed no indication of reaction with graphite and oxygen after a 1000-hr exposure. Weight measurements before and after the exposure show no change in weight for the Ir—2% Ru alloy and a weight loss of 46 ppm for Ir—3.84% W. The results of tensile tests of the exposed specimens are presented in Table 4 together with those for unexposed specimens. The

Table 4. Tensile Properties of Iridium Alloy Sheet Specimens 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		Elongation (%)
		Yield	Tensile	
		Room Temperature		
Ir—2 Ru	0	22.5	55.4	8.4
	1000	19.9	52.5	8.9
Ir—3.84 W	0	51.7	64.0	4.0
	1000	46.8	62.5	3.8
		760°C		
Ir—2 Ru	0	13.7	69.2	25.2
	1000	18.3	64.0	21.6
Ir—3.84 W	0	30.0	57.3	10.6
	1000	30.1	88.2	19.8
		1370°C		
Ir—2 Ru	0	9.0	32.8	52.3
	1000	10.3	32.4	46.5
Ir—3.84 W	0	21.7	46.9	25.1
	1000	22.2	44.0	27.3

data indicate that exposure to this simulated heat-source environment of oxygen and graphite at 1300°C did not impair the ductility or the strength of the alloys.

Air Oxidation of Pt-3008

H. Inouye

Short-term (to 200 hr) air oxidation rates of Pt-Rh-W alloys have been reported to be very low at 760, 1000, and 1200°C (ref. 10). To obtain long-term data, sheet specimens 0.020 × 0.62 × 1.0 in. of Pt-3008 (heat Pt-4V) are being oxidized for 10,000 hr at 600, 800, and 1000°C in still air. Because of the expected low oxidation rates, three specimens are being oxidized at each temperature to increase the reliability. The average weight change for an exposure of 283.4 hr is given in Table 5. No

Table 5. Air Oxidation of Pt–30% Rh–8% W (Pt-3008) in Still Air[a]

Test Temperature (°C)	Exposure (hr)	Oxidation Rate[b] ($g\ cm^{-2}\ hr^{-1}$)	Total Weight Change[b] (g/cm^2)
600	283.4	-4.9×10^{-8}	-1.4×10^{-5}
800	283.4	$+3.3 \times 10^{-7}$	$+9.4 \times 10^{-5}$
1000	283.4	$+1.8 \times 10^{-7}$	$+5.2 \times 10^{-5}$

[a]Specimen dimensions: 0.020 × 0.62 × 1.0 in.

[b]Average of three test specimens at each temperature.

visible evidence of oxidation was apparent for the small weight loss observed at 600°C. The specimens gained weight at 800°C and a black film was present on the surface. At 1000°C, the specimens gained weight (no visible oxide film) in contrast to our previous results which showed a

[10]H. Inouye et al., *New Pt-Rh-W Alloys for Space Isotopic Heat Sources*, ORNL-4813 (September 1972).

weight loss.[10] Small differences in the test conditions may account for the different results. In all respects, however, the low oxidation rates previously noted are confirmed.

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