**Isotopic Power Materials Development
Quarterly Progress Report for
Period Ending June 30, 1976**

A. C. Schaffhauser

OAK RIDGE NATIONAL LABORATORY

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METALS AND CERAMICS DIVISION

ISOTOPIC POWER MATERIALS DEVELOPMENT QUARTERLY PROGRESS
REPORT FOR PERIOD ENDING JUNE 30, 1976

A. C. Schaffhauser

Date Published: September 1976

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FOREWORD

This report is the third in a series of quarterly reports for Technology and Space Applications materials programs conducted by the Metals and Ceramics Division of Oak Ridge National Laboratory for the Nuclear Research and Applications Division of ERDA. These quarterly reports replace the monthly and annual reports previously issued on this work. The quarterly reports will provide a more detailed technical discussion of work in progress than was possible in the monthly reports. Significant technical highlights are reported to ERDA and their contractors on a monthly basis in the *Technical Highlights of Isotopic Power Materials Development at Oak Ridge National Laboratory* results, issued as CF memo reports, with limited distribution.

This quarterly report contains four chapters: (1) "High-Temperature Alloys for Space Isotopic Heat Sources," Activity No. KJ 30 01 02 3, 189a No. 00001; (2) "Physical and Mechanical Metallurgy of Heat-Source Containment Materials," Activity No. KJ 30 01 02 3, 189a No. 00002; (3) "Isotope Brayton System Materials Support," Activity No. KJ 30 01 02 2, 189a No. 00006; and (4) "Space Nuclear Flight Systems Hardware," Activity No. KJ 30 01 03 2, 189a No. 00003.

Recent previous reports covering work on these programs are listed below:

Isotopic Power Materials Development Quarterly Report

ORNL/TM-5285	Period Ending December 31, 1975
ORNL/TM-5482	Period Ending March 31, 1976

Isotopic Power Materials Annual Report

ORNL-TM-5116	Period Ending June 30, 1975
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Isotopic Power Materials Development Monthly Reports

ORNL-TM-4859	January 1975
ORNL-TM-4890	February 1975
ORNL-TM-4918	March 1975
ORNL-TM-4953	April 1975
ORNL-TM-4983	May 1975
ORNL-TM-5066	July 1975
ORNL-TM-5103	August 1975
ORNL-TM-5150	September 1975

Copies of these reports are available from the U.S. Energy Research and Development Administration, Technical Information Center, P.O. Box 62, Oak Ridge, Tennessee 37830.

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SUMMARY

1. HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES

Optimization of dopant additions to improve the high-temperature mechanical properties of Ir-0.3% W alloys was continued. Tensile tests at 1370°C show that the yield and tensile strengths increase with increasing thorium content between 0-500 ppm while maintaining greater than 50% elongation. Aluminum additions also appear beneficial to the high-temperature properties, and alloys containing both thorium and aluminum additions have been prepared. Additions of Fe, Ni, and Rh up to 80 ppm have no effect on the properties.

Doped iridium alloys containing 2% Ru are also being evaluated in an attempt to gain a further improvement in properties. Tensile tests at 650 and 760°C show a less ductile fracture mode at 650°C for alloys containing 2% Ru as compared to alloys without ruthenium. However, the alloys containing 2% Ru had a smaller grain size after annealing at 1500°C.

Characterization of the first Pt-3008 scale-up ingot was continued. Recrystallization studies (1-hr heat treatments) showed no recrystallization up to 950°C and complete recrystallization with a large drop in hardness at 1050°C. At 1000°C, the microstructure was partially recrystallized with some differences in the amount of recrystallization and hardness depending on sample location (nose, center, or tail of extrusion) and amount of cold finish rolling (40-50%). Tensile tests at 760 and 1093°C on sheet specimens rolled from different sections of the extrusion showed slightly higher yield strengths and lower ductilities on specimens from the center of the extrusion as compared to specimens from the nose and tail sections. Evaluation of the room-temperature mechanical properties of electron-beam welds in the scale-up heat shows that the yield strength of the welds is equivalent to annealed base metal; however, the maximum weld ductility is 4% at room temperature for a post-weld heat treatment of 1 hr at 1200°C.

Ruthenium-platinum and rhodium-ruthenium alloys are being evaluated as potential lower-density and/or lower-cost isotope fuel cladding materials for space applications. Experiments show that greater than 20 at. % Pt is

required to obtain a face-centered cubic (fcc) structure necessary for good fabricability. A Rh-20% Ru alloy had good fabricability as compared to pure rhodium, and ductility was increased by annealing at 900°C.

2. PHYSICAL AND MECHANICAL METALLURGY OF HEAT-SOURCE CONTAINMENT MATERIALS

Tensile impact tests at 1350°C and 85 m/sec (280 fps) were performed on a number of developmental Ir-0.3% W alloys after severe temperature exposures at 1500 and 1800°C to isolate and optimize the dopant concentrations for maximum ductility. The impact ductility increases linearly from <10% to >25% with thorium additions up to 200 ppm and plateaus between 200 and 1000 ppm. At the 30 ppm Th level, aluminum additions of 40 ppm are also effective in increasing impact ductility to >15%. At impact temperatures below 1200°C, the ductility of all alloys is severely reduced; however, the 200 ppm Th alloy was the most ductile alloy (15% elongation) at 950°C. Impact tests of specimens aged 2000 hr at 1330°C continue to show the improved ductility (24 vs 8%) of the DOP-4 alloy as compared to undoped Ir-0.3% W.

Auger analysis of Ir-0.3% W alloys shows that the grain boundary concentration of thorium is constant at 3-5 at. % for alloys containing 5-1000 ppm Th. Alloying with 2% Ru does not change this grain boundary concentration of thorium.

Tensile specimens of Pt-3008 oxidized 6000 hr continue to show the same effects as observed after 3000 hr. No effect on properties is observed at a 600°C exposure. At 800 and 1000°C exposures, the ductility at the exposure temperature is reduced to less than 2.5% due to formation of tungsten oxide phases in the grain boundaries.

3. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT

The mechanical properties of C-103 exposed to a simulated Brayton system atmosphere containing 1.3×10^{-3} Pa (1×10^{-5} torr) CO at 1000°C were determined. The creep rate at 1000°C and 48.2 MPa (7 ksi) stress was reduced in the carbon monoxide atmosphere as compared to the creep observed in ultrahigh vacuum. The specimen gained about 2000 ppm O and

1400 ppm C during the 300-hr test. The room-temperature tensile ductility after the creep test was reduced to 4.3%. Tensile tests at 25, 850, and 1000°C of specimens exposed to the same atmosphere for 200 hr showed a reduction of ductility to about 2.5% as compared to 20–30% for control specimens. Creep tests of alternate alloys for the Brayton system show that at 1000°C, molybdenum-rhenium alloys are equivalent or stronger than C-103 whereas the Pt-3008 alloy is much weaker.

Tensile specimens of Hastelloy X brazed to itself with Palniro 7 braze alloy and Hastelloy X brazed to C-103 with Coast Metals 50 braze alloy have been prepared for testing and aging.

4. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE

A total of 126 forming disks of doped Ir–0.3% W alloy and 1000 cm² of foil were fabricated and certified for MHW heat sources. The deliveries completed the requirements for the MJS mission and meet the requested schedule for the initial disks for the HPG/Mod-3 mission. The surface microdelaminations observed in some samples from the previous forming disks were determined to be an artifact of metallographic specimen preparation and they were eliminated by using a different specimen mounting material.

Additional tensile impact tests of HD-series production forming disks show impact ductilities of 19 to 20%.

1. HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES*

1.1 Introduction

The objective of this program is to develop and qualify improved alloys for encapsulation of $^{238}\text{PuO}_2$ isotope fuels used for heat sources for high-temperature energy conversion systems for space and terrestrial power applications. We are developing a series of high-melting-point, noble-base alloys having the required mechanical properties, oxidation resistance, and stability in a heat-source environment under both operational and potential severe accident conditions. Our activities are divided between improved iridium alloys, Pt-Rh-W alloys, and lighter-weight ruthenium and rhodium alloys. Tasks on these materials include alloy and fabrication development, characterization, and supply of materials and hardware for qualification testing.

1.2 Iridium Alloys

1.2.1 Development of Improved Ir-0.3% W Alloys — C. T. Liu and H. Inouye

We have doped Ir-0.3% W alloys with different levels of thorium to improve the metallurgical and mechanical properties. The tensile properties of DOP-21 and -22 were determined at 1370°C [at a crosshead speed of 5.1 mm/min (0.2 in./min)] after a standard heat treatment of 1 hr at 1500°C. The tensile data are presented in Table 1.1 and are compared with WTh-1 and undoped WG. The thorium content in these alloys is also listed in Table 1.1. The yield and tensile strengths increase with thorium content. All the alloys had more than 50% elongation and showed ductile rupture at 1370°C.

The microstructures of the DOP-21 alloy after short-term heat treatments at 1500 and 1800°C are shown in Fig. 1.1(a), (b), and (c). The heat-treated specimens show a recrystallized structure with second-phase particles (probably ThIr_5) within grains and on grain boundaries. The grain

*Progress on work performed under Activity No. KJ 30 01 02 3, 189a No. 00001.

Table 1.1. Tensile Properties^a of Doped and Undoped Ir-0.3% W
Sheet Specimens^b Tested at 1370°C

Alloy Specimen	Nominal Dopant Concentration (ppm)	Strength, MPa(ksi)		Elongation (%)	Fracture Mode ^c
		Yield	Tensile		
WG	None	38.6(5.6)	177.8(25.8)	55.2	DR
WTh-1	50 Th	48.2(7.0)	191.5(27.8)	66.1	DR
DOP-22	100 Th	58.6(8.5)	193.6(28.1)	53.8	DR
DOP-21	500 Th	73.0(10.6)	221.2(32.1)	56.0	DR
DOP-15	30 Th, 80 Fe	50.3(7.3)	188.1(27.3)	58.9	DR
DOP-13	30 Th, 40 Al	54.4(7.9)	188.1(27.3)	53.2	DR
DOP-16	30 Th, 80 Fe, 40 Al	54.4(7.9)	186.7(27.1)	55.3	DR
DOP-4(Ir-0.3% W)-10	30 Th, 80 Fe, 40 Al, 16 Ni, 75 Rh	55.8(8.1)	189.5(27.5)	53.6	DR

^aTested at a crosshead speed of 5.1 mm/min (0.2 in./min) in vacuum.

^bAll specimens annealed 1 hr at 1500°C before testing.

^cDR = ductile rupture.

Fig. 1.1 Microstructures of DOP-21 (500 ppm Th) Specimens Heat Treated (a) 1 hr at 1500°C, (b) 19 hr at 1500°C, and (c) 1 hr at 1500°C Plus 1 hr at 1800°C Plus 1 hr at 1500°C. 100×.

size of DOP-21 is comparable to WTh-2 (doped with 1000 ppm Th), but is finer than that of DOP-14 (doped with 200 ppm Th) under the same heat treatments. A comparison of the grain sizes of the thorium-doped alloys is shown on p. 20 of this report. The tensile impact properties of DOP-21 are reported in the "Physical and Mechanical Metallurgy of Heat-Source Containment Materials" chapter (chapter 2 of this report).

The published partial phase diagram of iridium-thorium system indicates¹ that thorium may lower the melting point of iridium. The eutectic temperature between iridium-rich solid solution and ThIr_5 (the first compound formed on the iridium-rich end) was estimated to be greater than 1500°C . To determine the melting point of the thorium-doped alloys, the specimens will be quickly heated to preset temperature and kept at that temperature for 10 min. The preliminary result indicates that DOP-14 (200 ppm Th) shows no indication of melting at 2000°C .

The tensile properties of the Ir-0.3% W alloys doped with DOP-4 dopants were determined at 1370°C . The results in Table 1.1 show that DOP-4 dopants do not affect the strength and ductility of DOP-4, -13, -15, and -16 at 1370°C . All the alloys fractured by ductile rupture with close to 100% reduction of area. The tensile impact properties of these alloys are reported in chapter 2 of this report.

The previous studies² indicate that doping with aluminum at a level of 40 ppm has a significant effect on grain structure and impact properties of Ir-0.3% W. To further characterize and optimize the aluminum effects, four new alloys, doped with aluminum and a combination of aluminum and thorium, were prepared by arc melting. The four alloys are (1) DOP-23 - Ir-0.3% W alloy doped with 40 ppm Al alone; (2) DOP-24 - Ir-0.3% W alloy doped with 30 ppm Th and 100 ppm Al; (3) DOP-25 - Ir-0.3% W alloy doped with 30 ppm Th and 200 ppm Al; and (4) DOP-4(Ir-0.3% W)-11 - skulls and heads of production material HD-460 and -461 with 40 ppm Al added (This ingot was prepared for studying the upgrading of recycled production material.). The four ingots were then fabricated into 0.76-mm- (0.030-in.-) thick sheets using the WGD production rolling schedule.³ The evaluation of their properties is in progress.

1.2.2 Development of Advanced Iridium Alloys with Dopants — C. T. Liu

Tensile properties of the two advanced alloys, DOP-17 (Ir-2% Ru-0.3% W) doped with 200 ppm Th) and DOP-18 (Ir-2% Ru-0.5% W doped with DOP-4 dopants), were determined at 650 and 760°C after a standard heat treatment of 1 hr at 1500°C. The results are presented in Table 1.2. The strength of DOP-17 is

Table 1.2. Tensile Properties^a of DOP-17 (Ir-2% Ru-0.3% W Doped with 200 ppm Th), DOP-18 (Ir-2% Ru-0.5% W Doped with DOP-4 Dopants) Alloy Specimens^b Tested at 650 and 760°C

Alloy Number	Strength, MPa(ksi)		Elongation (%)	Fracture Mode ^c
	Yield	Tensile		
<u>650°C</u>				
DOP-17	126.1(18.3)	670.4(97.3)	35.1	GBS (Ma) and TF (Mi)
DOP-18	98.5(14.3)	604.3(87.7)	36.5	GBS (Ma) and TF (Mi)
<u>760°C</u>				
DOP-17	125.4(18.2)	559.5(81.2)	41.1	Mainly TF
DOP-18	86.1(12.5)	485.8(70.5)	40.2	Mainly TF

^aTested at a crosshead speed of 2.54 mm/min (0.1 in./min) in vacuum.

^bAll specimens annealed 1 hr at 1500°C before testing.

^cGBS = grain-boundary separation; TF = transgranular fracture; Ma = major fraction; and Mi = minor fraction.

higher than that of DOP-18, but the ductility of both alloys is about the same. The DOP-17 and -18 alloys showed mixed fracture modes of grain-boundary separation (major fraction) and transgranular fracture (minor fraction) at 650°C while the Ir-0.3% W alloys doped with the same dopants exhibited² mainly transgranular fracture. These two alloys showed completely transgranular fracture at 760°C.

Figure 1.2 compares the microstructures of DOP-17 and -18. The specimens heat treated at 1500°C show a recrystallized structure with some second-phase particles within the grains and on grain boundaries. The DOP-17 alloy has finer grain structure than DOP-18 under the same heat treatment conditions. In terms of number of grains across the specimen thickness, DOP-17 had 27.8 and 21.6 as compared to 22.0 and 12.2 for DOP-18

Fig. 1.2. Microstructures of DOP-17 (Ir-2% Ru-0.3% W Doped with 200 ppm Th) and DOP-18 (Ir-2% Ru-0.5% W Doped with DOP-4 Dopants). (a) DOP-17 annealed 1 hr at 1500°C; (b) DOP-18 annealed 1 hr at 1500°C; (c) DOP-17 annealed 19 hr at 1500°C; and (d) DOP-18 annealed 19 hr at 1500°C.

after 1- and 19-hr annealing at 1500°C. The grain size of DOP-17 and -18 is finer than that of the Ir-0.3% W alloys doped with the same dopants.

1.3 Pt-Rh-W Alloys

1.3.1 Pt-3008 Scale-Up and Fabrication Development — M. M. Martin

Retention of a fibrous structure in fabricated Pt-3008 (Pt-30% Rh-8% W) sheet after stress relieving improves formability of this alloy. Figure 1.3 shows the effect of various heat treatments of 1-hr duration at temperatures in vacuum on hardness of cold-rolled material. As reported previously,⁴ the

Fig. 1.3. Effect of Heat-Treating Temperature on Diamond-Pyramid Hardness Number of Pt-3008 Sheet Fabricated from E1 Extruded Bar.

fabrication history for the specimens included extrusion from a consumable vacuum-arc-melted scale-up ingot followed by hot rolling and annealing at 1200°C (2192°F), and warm rolling and stress relieving at 1000°C (1832°F). Final sheet thickness was obtained by cold rolling. Specimens from the tail of the extrusion were final cold rolled 50% in thickness while those from the nose and middle were final cold rolled 40%.

The hardnesses of the cold-worked Pt-3008 materials (as shown in Fig. 1.3) decrease sharply with increasing temperature between 950 and 1050°C (1742 and 1922°F). This reduction of hardness is more pronounced for sheets that were reduced 50% than 40% at room temperature. The specimens with the higher percentage of cold work also exhibited maximal initial hardness of about 400–450 DPH, as revealed at the lower heat-treating temperatures in Fig. 1.3. At and above 1050°C (1922°F), however, all showed similar values of about $219\text{--}228 \pm 9$ DPH.

Figure 1.4 shows microstructures of three of the cold-worked specimens that were heat treated at 950, 1000, and 1050°C (1742, 1832, and 1922°F). These photomicrographs show all principal differences in structure from all heat treatments and form the basis to divide Fig. 1.3 into three distinct temperature regions. The resultant structure is either fibrous, partially recrystallized, or fully recrystallized. That is, all of the cold-worked specimens from nose, center, and tail of the extrusion retain their before-heat-treatment fibrous structure up to and between 900–950°C (1652–1742°F); they then begin to recrystallize either at or slightly above the higher temperature. After 1 hr at 1000°C (1832°F), the examination of specimens from the nose, center, and tail portions of the extrusion reveals structures in which about 25, 50, and 75%, respectively, contain equiaxed grains. These degrees of recrystallization are given in Fig. 1.3 in parentheses beside the corresponding plotted average hardness numbers. In the region between 950 and 1050°C (1742 and 1922°F) where partial recrystallization occurs, a lower hardness number apparently corresponds to an increase in area of equiaxed grains. At and above 1050°C (1922°F), all of the cold-worked specimens from nose, tail, and center of the extrusion exhibits a fully recrystallized structure of fairly equiaxed grains of ASTM grain size nos. 6–8.

To summarize the above results, Pt-3008 sheets fabricated from our first scale-up ingot with either 40 or 50% final cold rolling will

Fig. 1.4. Polished and Etched Specimens of Pt-3008 Sheets from Central Portion of Extruded Bar after Being Reduced Approximately 40% in Thickness at Room Temperature and then Heat Treated 1 hr in Vacuum: (a) 950°C (1742°F); (b) 1000°C (1832°F); and (c) 1050°C (1922°F). 100×.

recrystallize fully when heated in vacuum 1 hr at temperatures greater than or equal to 1050°C (1922°F). At lower stress-relieving temperatures of less than or equal to 950°C (1742°F) for 1 hr in vacuum, these sheets should retain their fibrous cold-worked structure.

Fabrication of additional Pt-3008 sheets for LASL and ORNL qualification studies and test hardware continues. The first scale-up ingot [approximately 60 mm (2 3/8 in.) in diameter, weighting 6 kg (13 lb)] was canned in a 4-in.-diam heavy-wall molybdenum shell and was extruded successfully (along with the molybdenum leader and TZM tail) into an oval-shaped bar. The second scale-up ingot [approximately 73 mm (2 7/8 in.) in diameter, weighing 8.4 kg (18.5 lb)] was coated with a thin layer of plasma-sprayed molybdenum to minimize ovality and increase yield. However, this second ingot did not extrude and developed numerous cracks when the molybdenum leader failed to extrude due to high hardness. Similar conditions were used for both extrusions; however, the second ingot was probably at lower temperature during upset due to less mass of molybdenum around the Pt-3008. The second ingot has been remelted for subsequent extrusion in a fully annealed heavy-wall molybdenum container.

A potential and perhaps more economical method of fabrication Pt-3008 sheet than extrusion is rolling an induction-melted casting. To investigate the feasibility of this method and to provide a commercial source for the alloy, a commercial company is casting a 19-mm- (0.75-in.-) thick ingot from 5.6 kg (12.4 lb) of ORNL's prealloyed material. This casting should be received for evaluation and rolling studies during July 1976.

1.3.2 Characterization of Pt-3008 Scale-Up Heat — H. Inouye

Sections from the nose, center, and tail of an extruded sheet bar of Pt-3008 were rolled to 0.5-mm- (0.020-in.-) thick sheet and tensile tested to determine whether the properties depended on the location. Specimens with their gage section parallel and transverse to the rolling direction were also tested to determine if any directionality of properties existed. The results listed in Table 1.3 show that the sheet prepared from the center of the extrusion [sheet E1(4B)] tends to have a slightly higher yield strength and lower ductility than either the nose or tail sections at 760

Table 1.3. Effect of Specimen Location and Orientation on Tensile Properties of Pt-3008 Sheet^a

Specimen Description		Strength, MPa(ksi)		Elongation (%)
Location in Extrusion	Orientation to Rolling Direction	Ultimate Tensile	0.2% Yield	
<u>25°C</u>				
Nose	Parallel	601.5(87.3)	340.0(49.2)	11.0
Nose	Transverse	640.8(91.8)	341.7(49.6)	12.6
Center	Parallel	695.2(100.9)	348.6(50.6)	16.4
Tail	Parallel	646.3(93.8)	318.3(46.2)	16.0
Tail	Transverse	724.8(105.2)	326.6(47.4)	23.2
<u>760°C</u>				
Nose	Parallel	549.3(79.7)	161.2(23.4)	34.6
Nose	Transverse	569.8(82.7)	168.8(24.5)	35.2
Center	Parallel	547.8(79.5)	196.4(28.5)	28.2
Tail	Parallel	551.2(80.0)	148.8(21.6)	36.2
Tail	Transverse	534.0(77.5)	151.6(22.0)	35.4
<u>1093°C</u>				
Nose	Parallel	261.8(38.0)	128.8(18.7)	20.0
Nose	Transverse	291.5(42.3)	127.5(18.5)	36.2
Center	Parallel	241.8(35.1)	141.3(20.5)	14.6
Tail	Parallel	277.7(40.3)	130.2(18.9)	36.0
Tail	Transverse	281.1(40.8)	128.8(18.7)	40.8

^aHeat E1. Consumable arc melt, extruded to sheet bar and rolled to 0.020-in.- thick sheet. Annealed 1 hr at 1200°C.

and 1093°C. The tensile properties parallel or transverse to the rolling direction were essentially the same. The observed scatter in the properties is similar to that observed in the small drop-cast ingots.

1.3.3 Welding of Pt-3008 Alloy — G. M. Goodwin

Testing of electron-beam weldments in sheet from the first scale-up heat [sheet E1(5)] is continuing. To date, fifteen tensile tests have been performed, all at 20°C, as noted in Table 1.4.

Base metal stress relieved 1 hr at 850°C shows high strength at room temperature with 12% total elongation. Annealing 1 hr at 1200°C causes full recrystallization with the usual reductions in strength. Yield

Table 1.4. Room-Temperature Tensile Properties of Pt-3008
Electron-Beam Weldments^a

Specimen Designation	Specimen Type ^b	Strength, MPa(ksi)		Total Elongation (%)	Fracture Location
		0.2% Offset Yield	Ultimate		
E1(5)-1	B	1149.4(166.7)	1393.5(202.1)	12.0	—
E1(5)-2	B	1149.4(166.7)	1393.5(202.1)	12.0	—
E1(5)-5	B-R	317.2(46.0)	606.8(88.0)	c	c
E1(5)-13	B-R	330.4(47.9)	474.0(68.8)	5.0	—
E1(5)-14	B-R	330.4(47.9)	502.8(72.9)	6.5	—
E1(5)-3	W	430.9(62.5)	430.9(62.5)	2.0	weld
E1(5)-4	W	459.7(66.7)	459.7(66.7)	2.0	weld
E1(5)-6	W-P	331.0(48.0)	413.7(60.0)	3.2	weld
E1(5)-15	W-P	316.0(45.8)	402.2(58.3)	4.0	weld
E1(5)-7	R-W	303.4(44.0)	386.1(56.0)	2.0	base metal
E1(5)-8	R-W	275.8(40.0)	399.9(58.0)	2.0	base metal
E1(5)-9	R-W	399.9(58.0)	399.9(58.0)	2.0	weld
E1(5)-10	R-W	317.2(46.0)	317.2(46.0)	2.0	weld
E1(5)-11	R-W-P	303.4(44.0)	455.1(66.0)	4.0	weld
E1(5)-12	R-W-P	303.4(44.0)	427.5(62.0)	4.0	weld

^aFull-penetration welds in 0.56-mm (0.022-in.) sheet from heat E1-5, stress relieved 1 hr at 850°C prior to welding. All tests performed at 20°C.

^bB = base metal; R = reannealed 1 hr at 1200°C prior to welding; W = transverse electron-beam weld; and P = post-weld heat treated 1 hr at 1200°C.

^cFailed in specimen shoulder.

strength drops by a factor of 3 to 4 ultimate strength by a factor of 2 1/2; however, ductility is also decreased by a factor of 2 [specimens E1(5)-5, -13, and -14 vs E1(5)-1 and -2].

Electron-beam welds in as-stress-relieved sheet [specimens E1(5)-3 and -4] show yield and ultimate strength values less than half that of stress-relieved base metal. Fracture occurs in the weld with low ductility (2% total elongation). Post-weld annealing 1 hr at 1200°C gives the weldment strength properties comparable to annealed base metal and improves the ductility slightly [specimens E1(5)-6 and -15].

Welds in annealed sheet [specimens E1(5)-7, -8, -9, and -10] show strength properties similar to annealed base metal and low ductility (2% total elongation). The results from specimens E1(5)-7 and -8 are suspect since fracture occurred in the shoulder radius, but the low ductility is confirmed by specimens E1-9 and -10.

Post-weld annealing of welds in annealed sheet for 1 hr at 1200°C (E1-11 and -12) gives a slight ductility improvement as it did for welds in stress-relieved sheet.

1.4 Ruthenium and Rhodium Alloys

H. Inouye

This task is directed toward the identification and the characterization of suitable cladding materials for isotopic fuels with lower density and/or lower cost than iridium- or platinum-base alloys.

1.4.1 Ruthenium-Platinum Alloys

Table 1.5 lists the density, structure, and lattice constants of binary ruthenium-platinum alloys containing less than 20 at. % Pt. The close-packed hexagonal (cph) structure of ruthenium persists in alloys with 15 at. % Pt or less; however, a small amount of a face-centered cubic (fcc) phase was identified in the 20 at. % Pt alloy. This fcc phase with a lattice parameter of 3.86 Å is assumed to be a solid solution of ruthenium and platinum. Since the 20 at. % Pt alloy severely cracked on hot rolling, it appears that fabricable alloys in this system will require more platinum; however, as Table 1.5 shows, platinum increases the density.

1.4.2 Rhodium Alloys

An arc-drop-cast ingot of rhodium was readily hot rolled to 0.5-mm- (0.020-in.-) thick sheet. The final rolling temperature of 1000°C resulted in a sheet too brittle to prepare test specimens at room temperature. Rerolling the sheet at 800°C restored some ductility and may permit specimen preparation. In contrast, a Rh-20% Ru alloy (also rolled to sheet at 1000°C) was bent 25 deg over a 2T bend radius at room temperature. As

Table 1.5. Density and X-Ray Data for Ruthenium-Platinum
Alloys Annealed 1 hr at 1600°C

Composition (at. % Pt)	Density (g/cm ³)	Structure	Lattice Constants, Å		
			<i>a</i>	<i>b</i>	<i>c/a</i>
0	12.45	cph	2.6987	4.2730	1.5834
5	12.78	cph	2.7090	4.2916	1.5842
10	13.27	cph	2.7124	4.2989	1.5849
15	13.76	cph	2.7140	4.3065	1.5868
20	14.20	cph, fcc ^a	2.7163	4.3151	1.5886

^aThree weak peaks, $\alpha_0 = 3.86$ Å.

shown in Table 1.6, this bend angle can be increased to 78 deg after annealing 1 hr at 900°C.

The rhodium-ruthenium diagram has recently been reported to be a peritectic-type system with a narrow two-phase region between 40–60 at. %.⁵ Alloys containing over 20 at. % Ru could, therefore, be fabricable.

Table 1.6. Effect of Heat Treatment on 2T Bend Angles
of Rh–20% Ru at Room Temperature

1-hr Heat Treatment at (°C)	Bend Angle (deg)
As-rolled at 1000°C	25
400	48
600	51
700	57
800	62
900	78
1000	63
1100	35
1300	24

1.5 References

1. F. A. Shunk, *Constitution of Binary Alloys, Second Supplement*, McGraw Hill Book Company, New York (1969), p. 466.
2. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Mar. 31, 1976*, ORNL/TM-5482.
3. R. G. Donnelly, *Isotopic Power Materials Development Ann. Progr. Rep. June 30, 1975*, ORNL-TM-5116, p. 2.
4. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Mar. 31, 1976*, ORNL/TM-5482, pp. 13–20.
5. E. M. Savitskii, V. P. Polyakova, N. R. Rosman, "Interaction of Ruthenium with Platinum Group Metals," *All-Union Conference on the General Characteristics in the Construction of Structural Diagrams of Metallic Systems*, 5th, Moscow (1971), pp. 47–9.

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2. PHYSICAL AND MECHANICAL METALLURGY OF HEAT-SOURCE CONTAINMENT MATERIALS*

2.1 Introduction

The high reliability in long-term unattended operation, high operating temperatures, and potentially severe accident conditions of isotope-fueled energy conversion systems require that materials for critical components be exclusively tested and that the limitations on their use be defined. The objective of this program is to evaluate and determine the suitability and limitations of such materials. To accomplish this objective, we have concentrated our efforts on the evaluation of materials under the most severe potential accident conditions (e.g., high-velocity reentry and impact of an isotope heat source from space after long-term operation). The potential degradation of material properties from environmental and impurity effects is being evaluated along with studies on the mechanisms controlling the properties of materials of interest. In addition, special projects and consulting on materials problems for isotope-fueled energy conversion systems are performed as requested by ERDA.

2.2 Characterization of Impact Properties

2.2.1 Impact Properties of Iridium Alloys — C. T. Liu

2.2.1.1 DOP-4 Dopant Effect. — Tensile specimens of DOP-13, -15, and -16 alloys were heat treated at 1500 and 1800°C and then impact tested at 1350°C at a velocity of 85 m/sec (280 fps) to isolate the effects of DOP-4 dopants. The impact results are presented in Table 2.1 and are compared with DOP-4 alloy and undoped WG. The grain size of the impact specimens is also presented in Table 2.1. At the three heat-treatment conditions shown in Table 2.1, the DOP-13 alloy, doped with 30 ppm Th and 40 ppm Al, consistently showed finer grain structure and much better impact properties, as compared with the DOP-15 alloy doped with 30 ppm Th and 80 ppm Fe. This indicates that aluminum, in addition to thorium, is effective in improving

*Progress on work performed under Activity No. KJ 30 01 02 3, 189a No. 00002.

Table 2.1. Tensile Impact Properties of Ir-0.3% W Doped with DOP-4 Series of Dopants^a

Alloy Specimen	Nominal Dopant Concentration (ppm)	Grain Size ^b	Elongation (%)	Reduction of Area (%)	Fracture Mode ^c
<u>Annealed 1 hr at 1500°C</u>					
WG-202	None	10.7	12.6	28	Mainly GBS
DOP-15	80 Fe, 30 Th	15.6	31.2	92	DR
DOP-13	40 Al, 30 Th	23.9	40.4	95	DR
DOP-4	40 Al, 80 Fe, 30 Th, 16 Ni, 75 Rh	19.2	37.6	94	DR
<u>Annealed 19 hr at 1500°C</u>					
WG-202	None	5.8	10.7	24	Mainly GBS
DOP-15	80 Fe, 30 Th	8.1	15.1	39	TF and GBS
DOP-13	40 Al, 30 Th	12.2	27.2	64	TF and DR
DOP-16	40 Al, 80 Fe, 30 Th	10.3	26.6	56	TF and DR
DOP-4	40 Al, 80 Fe, 30 Th, 16 Ni, 75 Rh	9.9	26.8	60	TF and DR
<u>Annealed 1 hr at 1500°C + 1 hr at 1800°C + 1 hr at 1500°C</u>					
WG-202	None	2.4 ^d	3.4 ^d	6 ^d	Completely GBS
DOP-15	80 Fe, 30 Th	—	9.9	20	Mainly GBS
DOP-13	40 Al, 30 Th	5.7	15.7	32	GBS and TF
DOP-16	40 Al, 80 Fe, 30 Th	—	10.7	23	Mainly GBS
DOP-4	40 Al, 80 Fe, 30 Th, 16 Ni, 75 Rh	3.8	11.0	19	Mainly GBS

^aImpact condition: 1350°C and 85 m/sec (280 fps).

^bNumber of grains across a 0.64-mm- (0.025-in.-) thick sheet.

^cGBS = grain-boundary separation; TF = transgranular fracture; Ma = major fraction; Mi = minor fraction; and DR = ductile rupture.

^dAverage of two tests.

the impact properties of Ir-0.3% W alloy. The impact properties of DOP-16 and DOP-4 are about the same, indicating that nickel and rhodium have no major effect on improving impact properties of Ir-0.3% W. The DOP-13 alloy, which had 15.7% elongation* after a severe heat treatment at 1500–1800°C,

*With this ductility, the DOP-13 alloy may survive the MHW/FSA-type impact test.

has the best impact properties among these alloys. Thus, our isolation of the DOP-4 dopants leads to the conclusion that aluminum, in addition to thorium, improves the impact behavior of Ir-0.3% W.

2.2.1.2 Thorium Concentration Effect. — The tensile impact properties of Ir-0.3% W alloys doped with 500 ppm Th (DOP-21) and 50 ppm Th (WTh-1) were determined after heat treatments at 1500–1800°C. All the heat-treated specimens were impacted at a velocity of 85 m/sec (280 fps) at 1350°C. The results are presented in Table 2.2 and are compared with the other thorium-doped alloys reported previously.¹ The impact elongation is also plotted as a function of thorium content in Fig. 2.1. After a heat treatment of 1 hr at 1500°C, the impact elongation increases almost linearly with thorium content in the alloy to 100 ppm, but it becomes less effective above this level (Fig. 2.1). The alloys showed ductile rupture with more than 87% reduction of area after being doped with 50 ppm Th or more. After a heat treatment of 19 hr at 1500°C, the impact elongation and reduction of area also increase with thorium content and become less sensitive to thorium content in the range 200–1000 ppm. After a severe heat treatment at 1500 and 1800°C, the alloys doped with 200–1000 ppm Th had 25.2–26.6% elongation and 71–83% reduction of area and fractured by ductile rupture and transgranular fracture, which are distinctly better than the alloys doped with lower levels of thorium. Thus, the DOP-14 and -21 and WTh-2 are most resistant to degradation due to several heat treatments. The plot in Fig. 2.1 shows that the DOP-21 alloy, doped with 500 ppm Th, is slightly superior to DOP-14 and WTh-2 under all heat-treatment conditions.

2.2.1.3 Impact Temperature Effect. — The thorium-doped alloys were impacted at 950°C to show the impact temperature effect. All the specimens were heat treated 1 hr at 1500°C and tested at a velocity of 85 m/sec (280 fps). The impact results are presented in Table 2.3. The impact elongation and reduction of area at 950°C increase with thorium content to 200 ppm and then decrease. This variation in impact ductility cannot be simply correlated with grain size as shown in Table 2.3. However, a correlation seems to exist between the impact properties at 950°C and the fracture behavior¹ at a slow strain rate at 650°C. The DOP-14 and -22 alloys,

Table 2.2 Effects of Thorium Content and Heat Treatment on Tensile
Impact Properties of Ir-0.3% W Sheet Specimens
Tested at 85 m/sec (280 fps) and 1350°C

Alloy Number	Thorium Content (ppm)	Grain Size ^a	Elongation (%)	Reduction of Area (%)	Fracture Mode ^b
<u>Annealed 1 hr at 1500°C</u>					
WG-202	0	10.7	12.6	28	Mainly GBS
WTh-1	50	20.9	25.5	89	DR
DOP-22	100	19.3	38.2	87	DR
DOP-14	200	25.3	37.9	92	DR
DOP-21	500	27.7	41.7	93	DR
WTh-2	1000	27.3	39.4	87	DR
<u>Annealed 19 hr at 1500°C</u>					
WG-202	0	5.8	10.9	23	Mainly GBS
WG-202	0	5.8	10.5	24	Mainly GBS
WTh-1	50	9.5	14.7	34	Mainly GBS
DOP-22	100	10.8	23.8	50	TF
DOP-14	200	15.0	28.9	84	DR
DOP-14	200	15.0	29.1	90	DR
DOP-21	500	22.6	33.8	91	DR
WTh-2	1000	21.8	c	79	DR
WTh-2	1000	21.8	29.0	87	DR
<u>Annealed 1 hr at 1500°C + 1 hr at 1800°C + 1 hr at 1500°C</u>					
WG-202	0	2.4	2.2	7	Completely GBS
WG-202	0	2.4	4.6	5	Completely GBS
WTh-1	50	4.2	7.2	17	Mainly GBS
DOP-22	100	6.5	14.1	27	Mainly GBS
DOP-14	200	12.0	25.2	71	DR and TF
DOP-21	500	15.8	26.6	83	DR and TF
WTh-2	1000	14.9	25.4	73	DR and TF

^aNumber of grains across a 0.64-mm- (0.025-in.-) thick sheet.

^bGBS = grain-boundary separation; TF = transgranular fracture; Ma = major fraction; Mi = minor fraction; and DR = ductile rupture.

^cNot able to measure; gage section destroyed.

Fig. 2.1. Impact Elongation of the Thorium-Doped Ir-0.3% W Alloys Tested at 1350°C and 85 m/sec (280 fps).

Table 2.3. Tensile Impact Properties of Undoped and Thorium-Doped Ir-0.3% W Sheet Specimens^a Tested at 950°C and 85 m/sec (280 fps)

Alloy Number	Dopants (ppm)	Grain Size ^b	Elongation (%)	Reduction of Area (%)	Fracture Mode ^c
WG	None	10.7	4.5	12	Mainly GBS
WTh-1	50 Th	20.9	6.2	13	Mainly GBS
DOP-22	100 Th	19.3	11.4	23	GBS (Ma) and TF (Mi)
DOP-14	200 Th	25.3	14.5	36	TF and GBS
DOP-21	500 Th	27.7	7.1	17	Mainly GBS

^aAnnealed 1 hr at 1500°C before impact test.

^bNumber of grains across a 0.60-mm- (0.02-in.-) thick sheet.

^cGBS = grain-boundary separation; TF = transgranular fracture; Ma = major fraction; and Mi = minor fraction.

which showed mainly transgranular fracture at 650°C, have the best impact properties at 950°C. The DOP-21 and WTh-1 alloys, which showed mixed modes of grain-boundary separation and transgranular fracture at 650°C, rank next. The undoped WG, which fractured mainly by grain-boundary separation at 650°C, has the poorest impact ductility at 950°C.

The impact elongation of DOP-14 is plotted as a function of test temperature in Fig. 2.2 and is compared with DOP-4 and -13 and undoped WG. The impact ductility of DOP-14 decreases with test temperature below 1200°C; however, it remains better than the other alloys at all the test temperatures. The DOP-14 alloy, which had 14.5% elongation and 36% reduction of area at 950°C, appears to be most resistant to low-temperature impact among the doped alloys.

2.2.2 Effect of Long-Term Heat Treatment on Impact Properties of Ir-0.3% W Alloys — C. T. Liu and D. E. Harasyn

To study the effects of long-term heat treatments at the MHW fuel clad temperature on the physical and mechanical properties of Ir-0.3% W alloys,

Fig. 2.2. Plot of Impact Elongation as a Function of Impact Temperature for Ir-0.3% W Alloys WG, DOP-4, DOP-13, and DOP-14. (All specimens heat treated 1 hr at 1500°C prior to impact test.)

WG (undoped) and HD (doped with DOP-4 dopants) specimens were annealed at 1330°C for various time periods and then impacted at 1350°C and 85 m/sec (280 fps). Table 2.4 summarizes the impact data for the specimens annealed to 2000 hr. The impact elongation is also plotted as a logarithmic function of the annealing time in Fig. 2.3. The impact ductility of both alloys decreases with annealing time; this decrease is apparently due to grain growth as indicated in Table 2.4. The DOP-4 alloy had 24% elongation while WG had only 7.8% elongation after a 2000-hr anneal. Thus, DOP-4 is much more resistant to degradation due to long-term heat treatment at 1330°C.

Table 2.4. Effect of Long-Term Anneal at 1330°C on the Impact Properties of WG^a and HD^b Specimens Tested at 85 m/sec (280 fps) and 1350°C

Specimen	Grain Size ^c	Impact Elongation (%)	Reduction of Area (%)	Fracture Mode ^d
<u>1-hr Anneal^e</u>				
WG	19.7	18.7	27	GBS and TF
DOP-4	26.9	36.0	94	DR
<u>200-hr Anneal</u>				
WG	—	13.7	28	Mainly GBS
HD	—	—	—	—
<u>480-hr Anneal</u>				
WG	8.0	8.4	24	Mainly GBS
HD	21.5	26.5	90	DR
<u>980-hr Anneal</u>				
WG	7.5	8.6	20	Mainly GBS
HD	16.9	27.2	88	DR
<u>2000-hr Anneal</u>				
WG	4.7	7.8	19	Mainly GBS
HD	13.3	24.0	61	Mainly TF

^aUndoped Ir-0.3% W specimens.

^bIr-0.3% W specimens doped with DOP-4 dopants.

^cNumber of grains across a 0.64-mm (0.025-in.-) thick sheet.

^dGBS = grain-boundary separation; TF = transgranular fracture; and DR = ductile rupture.

^eAnneal 1 hr at 1300°C.

Fig. 2.3. Effect of Long-Term Annealing at 1330°C on Impact Elongation of WG and DOP-4.

2.3 Microanalysis Studies

C. L. White, L. Heatherly, Jr., and R. E. Clausing

2.3.1 Thorium Segregation to Grain Boundaries in Ir-0.3% W

Because thorium appears to significantly affect the grain boundary strength of Ir-0.3% W alloys, it is of interest to determine the minimum thorium level that will result in acceptable grain boundary strength. In order to accomplish this, four experimental Ir-0.3% W alloys have been prepared with dopant levels as indicated in Table 2.5. These alloys were electron-beam melted and drop cast using WG lot powder and the head and skull of WTh-2 (Ir-0.3% W plus 100 ppm Th) as a master alloy.

Table 2.5. Thorium Dopant Levels in Experimental Heats of Ir-0.3% W Alloys

Heat Designation	Thorium Dopant Level (ppm)
WTh-3	25
WTh-4	10
WTh-5	5
WTh-6	50

Samples of each of these alloys have been heat treated 1 hr at 1500°C, then analyzed using Auger electron spectroscopy (AES), to determine their grain boundary chemistry.* The ratio, R , of the thorium (67 eV) peak height to the iridium (54 eV) peak height is plotted as a function of thorium dopant level in Fig. 2.4. It is clear that heat WTh-5, doped with only 5 ppm Th, has approximately the same concentration of thorium on the grain boundaries as do alloys with 50 ppm Th (WTh-1 and WTh-6).

If bulk chemical analysis verifies the thorium dopant levels in these alloys, impact testing of samples having small grain size will be considered.

*See reference no. 2 for a description of the experimental procedure.

Fig. 2.4. Doping Level for Thorium (wt ppm).

If alloys with as little as 5 ppm Th should have acceptable impact properties (given a small grain size), then alloy development efforts could concentrate on modifications specifically designed to control grain growth.

2.3.2 Auger Electron Spectroscopy Analysis of DOP-17 and DOP-18

Samples of DOP-17 (Ir-2% Ru-0.3% W plus DOP-4 dopants) and DOP-18 (Ir-2% Ru-0.3% W plus 200 ppm Th) have been analyzed using AES. Both of these alloys had thorium segregated to the grain boundaries in approximately the same concentration as other thorium-doped alloys.³ In addition to thorium and iridium, there was some indication of ruthenium on the fracture surface; however, interference between carbon and ruthenium would make a positive identification difficult.

2.3.3 Phosphorus Segregation to Grain Boundaries in Ir-0.3% W

Because there is evidence that small concentrations of phosphorus in Ir-0.3% W may significantly degrade its high-temperature impact properties,⁴ an experimental heat of Ir-0.3% W (IrP-3), doped with approximately 20 ppm P, has been prepared by D. E. Harasyn. This alloy was arc melted and drop cast using a pressed and sintered Ir-2% P compact as a master alloy. Because of the high vapor pressure of phosphorus, the amount of phosphorus retained in the cast alloy is uncertain, and bulk chemical analysis has not yet been obtained.

Preliminary AES analysis of two samples* of IrP-3 indicate very little phosphorus on the fresh fracture surfaces of the samples. Additional samples from various locations in the sheet will be analyzed to verify these results.

2.4 Environmental and Impurity Effects

2.4.1 Pt-3008 Air Oxidation — H. Inouye

Oxidation tests of Pt-3008 tensile specimens in still air, scheduled for 10,000 hr, logged 6000 hr on June 17, 1976. Table 2.6 compares the tensile properties of oxidized specimens with the control and annealed

*Heat treated 1 hr at 1500°C.

Tensile Properties of Pt-3008 (Heat Pt-5C)
Oxidized in Still Air^a

Exposure Conditions			Strength, MPa(ksi)		Elongation (%)	Reduction of Area (%)
Time (hr)	Temperature (°C)	Environment	Ultimate	0.2% Yield		
<u>600°C</u>						
1	1200	Vac (Annealed)	594.6(86.3)	198.4(28.8)	34.8	~100
6024	600	Vac (Control)	630.4(91.5)	228.1(33.1)	32.5	79
6024	600	Air	573.9(83.3)	208.1(30.2)	28.1	40
<u>800°C</u>						
1	1200	Vac (Annealed)	540.2(78.4)	182.6(26.5)	28.5	78
6024	800	Vac (Control)	470.6(68.3)	223.2(32.4)	26.1	38
6024	800	Air	153.6(22.3)	153.6(22.3)	1.3	~0
<u>1000°C</u>						
1	1200	Vac (Annealed)	311.4(45.2)	170.2(24.7)	13.2	12
6000	1000	Vac (Control)	304.5(44.2)	113.0(16.4)	32.7	44
5986	1000	Air	110.2(16.0)	97.1(14.1)	2.5	~0

^aSpecimen gage dimensions: 12.7 × 3.175 × 0.635 mm (0.5 × 0.125 × 0.025 in.); strain rate: 0.2/min.

specimens. The tensile properties of oxidized Pt-3008, as well as the control specimens after 6000 hr, are comparable to those measured after 3000 hr at all test temperatures. As noted previously and in the present test oxidation, temperatures of 600°C continue to have virtually no effect on the mechanical properties of the alloy; however, the alloy is embrittled by oxidation at 800 and 1000°C.

The microstructures of the embrittled alloy after 3042 hr at 1000°C in air are shown in Fig. 2.5. A coarse oxide phase is observed in the central regions of the specimen and a finely dispersed phase forms closer to the edges. Electron-microprobe examination shows that both the coarse and fine oxide phases are predominantly tungsten oxides.

2.4.2 Grain Size Effects in Pt-3008 — H. Inouye

The tensile properties of Pt-3008 have been reported to degrade after exposure to PuO₂ or to graphite at high temperatures; however, no evidence of a PuO₂ or graphite interaction has been found.^{5,6} Therefore, the reason for the loss in strength and ductility of compatibility specimens could be due to grain growth or contamination of the alloy by impurities.

Fig. 2.5. As-Polished Pt-3008 Oxidized 3042 hr in Still Air. Both coarse and fine dark phases are tungsten oxides. 150 $\times$.

To determine the effect of grain size on the tensile properties, specimens were annealed at 1200 and 1500°C for times of 1 and 16 hr in a noncontaminating environment. A summary of the results in Table 2.7 show (a) that both the ultimate and yield strengths decrease significantly with an increase in either the annealing temperature or the annealing time; (b) the ductility at 25°C is reduced to only 2.4% by a 16-hr anneal at 1500°C; however this heat treatment increases the ductility at 760, 900, and 1093°C. Thus, these results clearly show that grain size has a large effect on the tensile properties of the alloy and is likely the major reason for the observed properties of long-term compatibility specimens.

2.5 References

1. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Mar. 31, 1976*, ORNL/TM-5482.

Table 2.8. Effect of Heat Treatment on Tensile Properties of Pt-3008 (Heat E1-4B)

Heat Treatment	Strength, MPa(ksi)		Elongation (%)
	Ultimate	0.2% Yield	
<u>25°C</u>			
a	695.2(100.9)	348.6(50.6)	16.4
b	437.5(63.5)	219.1(31.8)	16.2
c	216.4(31.4)	195.7(28.4)	2.4
<u>760°C</u>			
a	547.8(79.5)	196.4(28.5)	28.2
b	446.5(64.8)	101.3(14.7)	45.0
c	356.2(51.7)	81.3(11.8)	42.8
<u>900°C</u>			
a	423.0(61.4)	168.1(24.4)	22.4
b	340.4(49.4)	91.6(13.3)	38.6
c	274.2(39.8)	73.7(10.7)	35.7
<u>1093°C</u>			
a	241.8(35.1)	141.3(20.5)	14.6
b	171.6(24.9)	71.0(10.3)	17.6
c	113.7(16.5)	62.0(9.0)	16.0

^aHeat treatment: 1 hr at 1200°C.

^bHeat treatment: 1 hr at 1200°C plus 1 hr at 1500°C plus 1 hr at 1200°C.

^cHeat treatment: 1 hr at 1200°C plus 16 hr at 1500°C plus 1 hr at 1200°C.

2. *Ibid*, p. 27.
3. *Ibid*, pp. 43-50.
4. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr Rep. Dec. 31, 1975*, pp. 41-51.
5. D. L. Rphr and S. S. Hecker, *Fracture Behavior of a Pt-30% Rh-8% W Alloy Between Room Temperature and 1300°C*, LA-UR-75-1189 (July 1975).
6. J. D. Watrous, *Plutonia/Curia Compatibility Testing Monthly Report for July 1974*, Donald W. Douglas Laboratories, Richland, Washington.

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3. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT*

3.1 Introduction

The objective of this program is to characterize the metallurgical and mechanical properties of materials and components selected for the Brayton Isotope Power System (BIPS) under the environmental and operating conditions expected for this system. Currently, emphasis is on the potential materials problems identified in the BIPS Ground Demonstration System (GDS), scheduled for operation in 1977. These potential materials problems include the effect of contaminants on the creep properties and tensile ductility of C-103 (Nb-10% Hf-1% Ti-0.7% Zr) in the heat exchanger, ducting, and turbine plenum; effects of aging and thermal cycling on the properties of C-103/Hastelloy X bimetallic joints; and the mechanical properties of braze alloys used for fabrication of the Hastelloy X recuperator. The magnitude of these potential materials problems and possible solutions are being investigated. Characterization of other key components including ducting and bellows will be performed. Additional tasks such as investigation of alternate materials and long-term creep tests will be initiated as required to insure the reliability of a flight system, scheduled for 1981.

3.2 C-103 Gaseous Interactions

H. Inouye and C. T. Liu

3.2.1 Creep Properties of C-103 in Contaminating Environments

The creep strength of C-103 has been measured in ultrahigh vacuum¹ but not in the BIPS environment, which is expected to be both oxidizing and carburizing in the temperature range 700-1000°C. A test just completed and those planned for the near future attempt to characterize the effects of specific gaseous impurities and effluents outgassed from system components on the creep behavior of the alloy.

A specimen was creep tested at 48.2 MPa (7 ksi) and 1000°C in vacuum of 10^{-7} to establish base-line data followed by testing the same specimen

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in 1.3×10^{-3} Pa (1×10^{-5} torr) CO. A rectangular sheet specimen was also exposed to carbon monoxide in a separate apparatus at the same conditions because dilation has been found to be one of the important effects of the contamination of tantalum.² Creep data based on tests of about 300 hr (Table 3.1) shows that C-103 creeps slower in carbon monoxide than in vacuum. Because dilation of the unstressed specimen was about 40% of the total creep strain measured in carbon monoxide at 48.2 MPa (7 ksi), the creep rate of C-103 that can be attributed to stress is calculated to be about one-third of the rate in vacuum. A post mortem of the creep specimen (Table 3.2) confirms the expected contamination of the alloy with carbon and oxygen, its embrittlement, and the protective effect of getter foils.

Table 3.1. Effect of the Environment on the Creep Rate of C-103 at 1000°C^a

Environment	Stress [MPa(ksi)]	Creep Rate (%/1000 hr)	Test Duration (hr)
Vacuum (1.3×10^{-5} Pa)	48.2(7)	1.4	300
CO (1.3×10^{-3} Pa)	48.2(7)	0.7	300
CO (1.3×10^{-3} Pa)	None (dilation)	0.3	140
Calculated Net Creep Rate ^c	—	0.4	—

^aGage dimensions: $0.762 \times 3.175 \times 50.8$ mm ($0.030 \times 0.125 \times 2.0$ in.).

^bConversion: 1.3×10^{-5} Pa = 1×10^{-7} torr; 1.3×10^{-3} Pa = 1×10^{-5} torr.

^cCreep rate due to stress is the difference between stressed and unstressed specimen in carbon monoxide.

3.2.2 Effect of Gaseous Environment on Embrittlement of C-103

To study the embrittlement effects of the carbon monoxide gas environment, tensile sheet specimens of C-103 were vacuum annealed 1 hr at 1650°C and then exposed to carbon monoxide gas at a pressure of 1.3×10^{-3} Pa (1×10^{-5} torr) at 1000°C. The specimens showed almost no indication of surface films up to 200-hr exposure. The average weight gain for the tensile specimens is 18 ppm/hr [in terms of 0.762-mm- (0.030-in.-) thick specimens]. The tensile properties of the annealed and exposed specimens

Table 3.2. Post Mortem of C-103 Creep Tested in
 1.3×10^{-3} Pa (1×10^{-5} torr) CO at 1000°C^a

Tensile Properties at 25°C	Strength, MPa(ksi)		Elongation (% in 2 in.)
	Ultimate	0.2% Yield	
Initial	418.2(60.7)	314.2(45.6)	29.5
Post Creep	456.8(66.3)	336.2(48.8)	4.3
Chemical Analyses	Oxygen (ppm)	Carbon (ppm)	$\Delta o/\Delta c$
Initial	135	181	—
Gage Section	2084 ($\Delta o = 1949$)	1570 ($\Delta c = 1389$)	
Shoulder ^b	533 ($\Delta o = 398$)	160 ($\Delta c = -21$)	—

^aThree hundred hours in 1.3×10^{-5} Pa (1×10^{-7} torr) vacuum plus 300 hr in 1.3×10^{-3} MPa (1×10^{-5} torr) CO; specimen thickness: 0.762 mm (0.030 in.).

^bProtected by 0.127-mm- (0.005-in.-) thick foil of Nb-1% Zr spot welded to both sides of specimen shoulder.

were determined at room temperature, 800°C, and 1000°C; the results are presented in Table 3.3 and plotted as a function of weight gain in Fig. 3.1.

Table 3.3. Tensile Properties of C-103 Sheet Specimens^a Exposed
to Carbon Monoxide Gas at 1000°C and 1.3×10^{-3} Pa
(1×10^{-5} torr) Pressure

Weight Gain (ppm)	Elongation (%)	Strength, MPa(ksi)	
		Yield	Tensile
<u>Room Temperature</u>			
0	29.5	314.2(45.6)	418.2(60.7)
1816 ^b	14.5	319.0(46.3)	420.3(61.0)
3928 ^c	2.3	335.5(48.7)	397.5(57.7)
<u>850°C</u>			
0	22.1	139.2(20.2)	287.3(41.7)
2052 ^b	7.4	212.2(30.8)	303.2(44.0)
4205 ^c	2.5	283.2(41.1)	306.6(44.5)
<u>1000°C</u>			
0	20.5	137.8(20.0)	281.1(40.8)
2107 ^b	10.0	210.1(30.5)	288.7(41.9)
4124 ^c	2.3	267.3(38.8)	305.9(44.4)

^aSpecimen thickness: 0.63–0.69 mm (0.025–0.027 in.).

^bExposed for 96 hr.

^cExposed for 196.65 hr.

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Fig. 3.1. Plot of Tensile Data as a Function of Weight Gain for C-103 Specimens Exposed to Carbon Monoxide Gas at 1000°C at a Pressure of 1.3×10^{-3} Pa (1×10^{-5} torr).

The yield strength at 850 and 1000°C increases linearly with weight gain, but the yield strength at room temperature and the tensile strength at the three test temperatures are relatively insensitive to the weight gain. The tensile ductility of the C-103 specimens decreases seriously with increasing weight gain or exposure time at 1000°C. The elongation of C-103 specimens was reduced from 20–30% to 2.3–2.5% at these three test temperatures after approximately 200 hr of exposure (weight gain $\approx$ 4000 ppm) at 1000°C.

The specimen which showed a weight gain of 3928 ppm was sent to vacuum fusion and carbon analyses. The result indicates that this specimen absorbed 1627 ppm C and 2315 ppm O, a total of 3942 ppm. The carbon-to-oxygen ratio is 1.42, which agrees reasonably well with a value of 1.5, reported³ for exposure of tantalum to carbon monoxide gas around 1000°C.

3.3 Alternate Alloys

H. Inouye and C. T. Liu

3.3.1 Creep Properties of Molybdenum-Rhenium Alloys

We characterize the creep properties of molybdenum-rhenium alloys in order to consider them as a back-up material for the Isotope Brayton System. Sheet specimens of Mo–45% Re and Mo–33% Re alloys, prepared by electron-beam melting, were tested in creep at 1000°C at a stress of 96.5 MPa (14 ksi) in a vacuum of 1.3×10^{-4} Pa (1×10^{-6} torr). The 1000-hr creep tests indicate that the creep strengths (in terms of time for 1% creep strain) of C-103 and Mo–45% Re are comparable to each other at 1000°C. The Mo–33% Re alloy is much stronger in creep and its creep rate is lower than that of Mo–45% Re and C-103 by a factor of 55.

3.3.2 Creep Properties of Pt-3008

The creep tests of Pt-3008 at 1000°C in vacua of approximately 1.3×10^{-4} Pa (1×10^{-6} torr) are in progress. The data show that the time required to produce 1% strain at 34.5 MPa (5 ksi) is 136.9 hr and 48.6 hr at 48.2 MPa (7 ksi). The time required to produce 1% strain in C-103 at 48.2 MPa (7 ksi) and 1000°C is reported to be about 1000 hr.

3.4 Welding and Brazing Evaluation

G. M. Goodwin

Lap-shear specimens have been prepared with Hastelloy X brazed to itself with Palniro 7 (Au-8% Pd-22% Ni). A joint spacing of 0.0762 mm (0.003 in.) was used, controlled both by nickel wire spacers and the braze alloy foil itself. After placement of the spacers, the specimens were autogeneously tack welded prior to brazing.

Dissimilar metal lap-shear specimens have also been prepared between Hastelloy X and C-103 alloy sheet using Coast Metals 50 (Ni-3.5% Si-2.5% B-1% Fe) braze alloy in powder form. Spacing was accomplished with nickel wires, and tack welds were made with Inconel 82 filler metal due to the extreme brittleness of the direct fusion weld.

Both sets of specimens are being machined for short-term tensile tests and aging studies.

3.5 References

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2. J. B. Conway, R. M. Fincel, Jr., and A. C. Losekamp, *Trans. AIME* 233 (April 1965), p. 841-2.
3. M. D. Ketchum and C. A. Barrett, *Contamination of Refractory Metals by Oxygen, Water Vapor, and Carbon Monoxide*, NASA-CR-1537 (May 1970).

4. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE*

4.1 Introduction

The limited-scale production of high-quality iridium alloy sheet and foil, based on ORNL-developed procedures, is performed in this task. Forming disks are machined from sheet and are supplied to Mound Laboratory for hot hydroforming into hemispheres which encapsulate the $^{238}\text{PuO}_2$ isotope fuel spheres and serve as the Post-Impact Containment Shells (PICS) of the Multi-Hundred Watt (MHW) Heat Source. Iridium alloy foil is also supplied for forming weld shields and decontamination covers during processing the PICS. Extensive quality assurance and characterization of material, along with a limited amount of process improvement development, is performed in this task.

4.2 Production of Ir-0.3% W Forming Disks and Foil

D. E. Harasyn

Production data on Ir-0.3% W forming disks produced in April, May, and June of 1976 are shown in Table 4.1. We completed our required shipment of forming disks for the MJS mission with the shipment of HD 451-456 and began production for the HPG/Mod-3. Our present schedule calls for shipment of 20 disks to Mound Laboratory every three weeks.

Approximately 1000 cm² (157 in.²) of Ir-0.3% W foil was shipped to Mound Laboratory this quarter. It is expected that this shipment will meet Mound Laboratory's foil requirements for the MJS mission. We have requested the approval of Mound Laboratory to change our procedures so that foil is no longer recrystallized before shipment. Recrystallized Ir-0.3% W is very soft and easily damaged, which makes packaging of the foil very difficult. In the cold-worked state, however, the foil is very hard and resistant to damage during handling and packaging.

Surface microdelaminations that have shown up on most of the PICS production metallography samples from HD 427-462 appear to be only an artifact

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Table 4.1. PICS Production Data

Ingot Number	Number Machined	Nonconformance Report ^a				Disks Shipped				Total Shipped		Date Shipped		
		Disk Number				1	2	3	4	Per Lot	Cumulative for Quarter			
		1	2	3	4									
HD-433	4	M	M	U,M	M	1	1		1	19	19	4/9/76		
-434	4	M	M	M	M	1	1	1	1			4/9/76		
-435	4	U,M	M	M	M		1	1	1			4/9/76		
-436	4	U	U					1	1			4/9/76		
-437	4				U	1	1	1				4/9/76		
-438	4	M	M	M	V,M	1	1	1	1			4/9/76		
-439	4	M	M	M	U,M	1	1	1				4/21/76		
-440	4	M	M	M	P,M	1	1	1				4/21/76		
-441	4	M	M	M	M	1	1	1	1			4/21/76		
-442	4	U,M	M	M	M		1	1	1			4/21/76		
-443	4	M	M	U,M	U,M	1	1			4/21/76				
-444	4	M	M	M	M	1	1	1	1	19	38	4/21/76		
-445	4					1	1	1	1			5/7/76		
-446	4	U		U			1		1			5/7/76		
-447	4					1	1	1	1			5/7/76		
-448	4					1	1	1	1			5/7/76		
-449	4					1	1	1	1			5/7/76		
-450	3			U		1	1					5/7/76		
-451	4	M	M	M	M	1	1	1	1			20	58	5/13/76
-452 ^b	4					1	1	1	1					5/13/76
-453 ^b	4	M	M	M	M	1	1	1	1					5/13/76
-454	4	M	M	M	M	1	1	1	1			5/13/76		
-455 ^b	4					1	1	1	1			5/13/76		
-456 ^b	4					1	1	1	1	24	82	5/13/76		
-457	4				U	1	1	1				5/26/76		
-458	4				V	1	1	1	1			5/26/76		
-459	4	U			U		1	1				5/26/76		
-460	4					1	1	1	1			5/26/76		
-461	4	M	M	M	M	1	1	1	1			5/26/76		
-462	4		U,D			1		1	1			5/26/76		
HDR-463	4					1	1	1	1			20	102	6/22/76
-464	4					1	1	1	1					6/22/76
-465	4					1	1	1	1					6/22/76
-466	4					1	1	1	1			6/22/76		
-467	4					1	1	1	1			6/22/76		
-468	4					1	1	1	1	24	126	6/22/76		

^aD = dimensional; U = ultrasonic; P = dye penetrant; V = visual; and M = metallography.

^bSintered compacts were cleaned because of surface contamination.

of the metallographic preparation. It has been demonstrated that a chemical or metallurgical difference in the lot HD 427-432 (first lot to show delaminations) from previous lots could not account for the delaminations.¹

A second experiment involved material that had been examined metallographically prior to HD 427-432 and had shown no delaminations. When this material was metallographically prepared some time after HD 427-432, it contained the surface delaminations. This led to an investigation of our metallographic procedure to determine the cause of the delaminations. A review of our data indicated that virtually any sample submitted after HD 427-432 that was surface ground and in the wrought condition was likely to be delaminated on the surface no matter what was done to it prior to the metallographic preparation.

A lack of edge support was thought to be the most likely cause of the surface delaminations. To establish this, we remounted and repolished a set of production samples from HD 439-444. All had failed the metallography inspection because of surface delaminations. The worst three of the six (HD-439, -440, and -444) were dissolved from their epoxy mounts and remounted in Plastimet (Buehler Ltd.). This new mounting material has a hardness of approximately 70 DPH as compared to the epoxy material which has a hardness of about 7 DPH. The new mounting material was advertised as having excellent edge-retaining qualities. The three samples that were remounted and repolished in Plastimet mounts contained no surface delaminations. All three of the samples that were left in their epoxy mounts and repolished at the same time with the samples mounted in Plastimet contained surface delaminations. These delaminations were severe enough to result in a failure of the metallographic certification. The edges of the samples mounted in Plastimet appeared very clean and straight, whereas the edges of samples mounted in epoxy appeared very rough, ragged, and sometimes cracked. Lack of edge support is thought to have been the most likely cause of the surface delaminations in all of our samples mounted in epoxy since HD 427-432. We suspect that the hardener for the epoxy was at fault, but the hardener is no longer traceable and further experiments are costly.

PICS production samples from HDR 463-468 were mounted in Plastimet, and none of these samples had evidence of surface delaminations. No experiments are planned to further investigate this problem because we believe our evidence pointing toward lack of edge support is solid. We intend to use Plastimet as the mounting material for all our production samples in the future.

4.3 Impact Properties of Production Material

C. T. Liu

We are continuing to qualify the impact properties of DOP-4-type Ir-0.3% W production material used for space nuclear flight systems hardware. Sheet specimens were blanked from heats HD-403, -436, -440, and -459, heat treated 19 hr at 1500°C, then impacted at 85 m/sec (280 fps) at 1350°C. The results are presented in Table 4.2 together with those obtained previously from different lots of production material. The production lots HD-403 to -459, produced by electron-beam melting of 40% powder compacts and 60% recycled heads and skulls from the proceeding lot of HD ingots, had the impact ductility of 15.8–20.8% which is generally lower than for lot DOP-4(0.3% W)-1 and HD-400 (produced from all powder compacts). As reported previously,² the lower ductility is probably due to loss of the aluminum dopants on successive electron-beam melts. Since the HD production material has more than 14% impact elongation, it should survive the MHW/FSA-type impact test.

4.4 References

1. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Mar. 31, 1976*, ORNL/TM-5482, p. 67.
2. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Mar. 31, 1976*, ORNL/TM-5482.

Table 4.2. Tensile Impact Properties of Ir-0.3% W Alloy Sheet
Annealed 19 hr at 1500°C and Tested at
85 m/sec (280 fps) at 1350°C

Heat Number	Grain Size ^a	Elongation (%)	Reduction of Area (%)	Fracture Mode ^b
WG-65 ^c	3.4	9.6	21	Mainly GBS
WG-204 ^c	3.8	10.7	23	Mainly GBS
DOP-4(0.3% W)-1 ^d	9.9	26.8	60	TF and DR
WGD-271 ^e	8.4	18.2	45	TF (Ma) and GBS (Mi)
HD-400 ^f	10.3	21.1	52	Mainly TF
HD-403 ^g	8.6	20.8	50	Mainly TF
HD-416 ^{g,h}	8.0	18.1	45	TF (Ma) and GBS (Mi)
HD-416 ^{g,h}	—	14.8	36	TF and GBS
HD-428 ^{g,h}	8.9	15.2	39	TF and GBS
HD-428 ^{g,h}	—	16.5	42	TF (Ma) and GBS (Mi)
HD-436 ^g	8.1	18.5	46	TF (Ma) and GBS (Mi)
HD-440 ^g	—	20.5	59	Mainly TF
HD-459 ^g	—	18.9	57	Mainly TF

^aNumber of grains across a 0.64-mm (0.025-in.) sheet thickness.

^bGBS = grain-boundary separation; TF = transgranular fracture; DR = ductile rupture; Ma = major fraction; and Mi = minor fraction.

^cUndoped Ir-0.3% W alloys.

^dIr-0.3% W alloy doped with DOP-4 dopants, produced from electron-beam melting of WG powder compacts.

^eIr-0.3% W alloy doped with DOP-4 dopants, produced from electron-beam melting of 40% WG powder compacts and 60% WG recycled skulls and heads.

^fIr-0.3% W alloy doped with DOP-4 dopants, produced from electron-beam melting of HD powder compacts.

^gIr-0.3% W alloy doped with DOP-4 dopants, produced from electron-beam melting of 40% HD powder compacts and 60% HD recycled skulls and heads.

^hDuplicate tests.

Blank

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