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Isotopic Power Materials Development Quarterly Progress Report for Period Ending December 31, 1976

A. C. Schaffhauser

OAK RIDGE NATIONAL LABORATORY

OPERATED BY UNION CARBIDE CORPORATION FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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METALS AND CERAMICS DIVISION

ISOTOPIC POWER MATERIALS DEVELOPMENT QUARTERLY PROGRESS
REPORT FOR PERIOD ENDING DECEMBER 31, 1976

A. C. Schaffhauser

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FOREWORD

This report is the fifth 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* reports, issued as CF memo reports, with limited distribution.

This quarterly report contains seven chapters: (1) "High-Temperature Alloys for Space Isotopic Heat Sources," Activity No. KJ 06 01 01 0, 189a No. 01405; (2) "Physical and Mechanical Metallurgy of Heat-Source Containment Materials," Activity No. KJ 06 01 01 0, 189a No. 01406; (3) "Curium-244 Fuel Development," Activity No. KJ 06 02 01 0, 189a No. 01367; (4) "Terrestrial Radioisotopes Applications Development," Activity No. KJ 06 01 00 0, 189a No. 01367; (5) "Selenide Isotope Generator System Support," Activity No. KJ 07 02 02 0, 189a No. 01320; (6) "Isotope Brayton System Materials Support," Activity No. KJ 07 02 01 0, 189a No. 01319; and (7) "Space Nuclear Flight Systems Hardware," Activity No. KJ 07 03 00 0, 189a No. 01322.

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

Isotopic Power Materials Development Quarterly Reports

ORNL/TM-5285	Period Ending December 31, 1975
ORNL/TM-5482	Period Ending March 31, 1976
ORNL/TM-5569	Period Ending June 30, 1976
ORNL/TM-5810	Period Ending September 30, 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 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 for improved iridium alloys for 1981-1983 flight missions was completed. Two alloy systems were selected for final characterization and qualification: arc-melted DOP-26 (Ir-0.3% W with additions of 60 ppm Th and 50 ppm Al), and alloys containing 200 ppm Th prepared either by arc melting (DOP-14-1 or DOP-27 also containing 50 ppm Al) or electron-beam melting (DOP-28). The tensile impact ductility at 1350°C and 85 m/sec (280 fps) of these alloys after the standard 19 hr at 1500°C thermal exposure was greater than 34% as compared to 15-23% for the current electron-beam-melted DOP-4 alloy production material and 10% for undoped material. After a severe thermal exposure of 1 hr at 1800°C, the DOP-14 alloy retained 25% ductility as compared to 13.5 for the DOP-26 alloy. However, preliminary weldability screening tests show that iridium alloys containing thorium levels of 100 ppm or greater are subject to weld centerline hot cracking. The weld cracking correlated with the quantity of precipitates in the fusion zone which increased with increasing thorium content and was greater than in the base metal. Based on these results and the qualification schedule requirements for an improved iridium alloy for the 1981 Improved Multihundred Watt (MHW) heat source and the 1983 General Purpose Heat Source (GPHS), the DOP-26 alloy was selected for fabrication scale-up. However, both the DOP-26- and DOP-14-type alloys will be supplied to LASL for fuel compatibility testing. If the weldability of the 200 ppm Th alloys can be improved, these alloys may be substituted at a later date to allow higher temperature exposures.

Fabrication and characterization of the second Pt-3008 consumable-arc-melted scale-up heat (E2X) was completed. Fabrication of a commercially induction-melted and cast ingot (IMPI-1) was also completed. The fabrication yield for these ingots was greater than 70%, and 8.7 kg (19.3 lb) of sheet were supplied for characterization and fabrication development. Tensile tests of sheet from the second scale-up heat after a 1-hr vacuum anneal at 1200°C showed the typical increase in ductility from 15% at room temperature to 30-40% at 150-900°C, a ductility minimum

of 15% at 1100°C, and increase in ductility to greater than 40% at 1316°C. Aging at 1100°C for 1000 hr in a static vacuum or 200 hr in hydrogen eliminated the ductility minimum in tensile tests at 1100°C.

Fabrication studies of arc-melted rhodium-ruthenium alloys show that alloys containing up to 45% Ru can be fabricated to plate. Alloys higher in ruthenium were not fabricable by conventional techniques due to their hexagonal crystal structure.

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

Tests to determine the effects of impact temperature and long-term high-temperature thermal exposures on the impact ductility of iridium alloys were continued. Tensile impact tests at 1050°C and 85 m/sec (280 fps) show that the ductility of all alloys was lower than at 1350°C. Increasing the annealing time from 1 to 19 hr at 1500°C significantly lowered the ductility at the low impact temperature. However, the DOP-14 and -26 alloys retained 18–20% ductility under the worse conditions compared to 9% for the DOP-4 alloy. Impact tests at 1350°C for specimens exposed up to 6000 hr at 1330°C in vacuum show that, due to its grain growth resistance, the DOP-14 alloy has about 3 times the impact ductility of the DOP-4 alloy (36% vs 17%). After the severe thermal exposure estimated for the emergency cooling condition for Brayton Isotope Power System (60 hr at 1660°C), the DOP-14 alloy retained 23% impact ductility.

Tensile tests of iridium alloys containing thorium additions exposed in air at 670 and 870°C for 1000 hr show no degradation in tensile properties. Metallographic examination of Pt-3008 exposed to graphite for 5 min at 1750–1950°C, simulating launch pad fire conditions, shows small spherical graphite particles in the matrix for the 1800°C exposure, but no indication of eutectic melting. At 1850°C and above, platelets of graphite were observed in the Pt-3008 matrix, probably due to localized eutectic reactions. An iridium layer between the Pt-3008 and graphite was not effective in reducing the interaction.

Extensive Auger analysis of DOP-4 iridium samples from ThO₂ simulant Fuel Sphere Assembly (FSA) impact tests shows that high levels of phosphorus contamination may have contributed to the failures observed in some

of these tests. Samples from FSA impacts MHT-103 and -104, which showed internal cracks and breaches, had three times the phosphorus contamination near the internal surface compared to samples from the successful FSA impacts MHT-90, -91, and -92. The Auger peak for thorium was also significantly reduced in the regions of highest phosphorus contamination. Analysis of grain boundary precipitates in an iridium alloy containing 1000 ppm Th, using a fine-beam scanning Auger microprobe, showed that the precipitate-matrix interface is about three times higher in thorium than the grain boundary.

3. CURIUM-244 FUEL DEVELOPMENT

Preparation for compatibility testing of $^{244}\text{Cm}_2\text{O}_3$ with tensile specimens of potential cladding materials has been resumed. The cladding materials and capsules were fabricated previously; however, three new alloys will be included in the revised test matrix. An extrusion technique for fabrication of the rectangular $^{244}\text{Cm}_2\text{O}_3$ fuel specimens appears to be a promising alternative to the previously unsuccessful hot pressing techniques.

4. SELENIDE ISOTOPE GENERATOR SYSTEM SUPPORT

Pt-3008 sheet for the bellows of the Isolation Hot Frame (IHF) of the Selenide Isotope Generator (SIG) ground demonstration system has been supplied to a commercial bellows fabricator for forming development. Fabrication of the required tooling for forming has been completed.

The initial compatibility screening tests of Pt-3008 with potentially incompatible materials in the system were completed for 1000 hr at 800, 950, and 1100°C exposures under the "worse case" conditions. No degradation of mechanical properties was observed in Pt-3008 samples in the vicinity of Min-K insulation in contact with graphite. Specimens in direct contact with Fiberfrax insulation had less than half the ductility of control specimens. Auger analysis of the grain boundary fracture surface showed 1-5 at. % S and P. Specimens exposed to vapor from Cu_2Se were completely brittle due to grain boundary corrosion and embrittlement from Se. A second series of tests under more typical conditions is being prepared.

We completed an assessment of commercially available fibrous thermal insulations of interest for the SIG system. Recommendations of additional data required to insure successful utilization are given. Chemical and microstructural characterization of the insulations currently proposed for the GDS show potential chemical and high-temperature stability problems.

5. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT

Environmental creep testing of C-103 at 878°C and 145 MPa (21 ksi) in a simulated alternator outgassing environment of pyromellitic acid showed a creep rate a factor of 10 higher than in ultrahigh vacuum. Initial parametric tests to determine the factors controlling the accelerated creep were unsuccessful due either to specimen bending or synergistic interactions between the stress and environment. Contamination kinetics studies on C-103 in 1.3×10^{-3} Pa (10^{-5} torr) CO show a square root of time dependence at 850 and 1000°C. The contamination rate is diffusion controlled and decreases with decreasing temperature and increasing specimen thickness. The tensile ductility of C-103 specimens contaminated to 4200 ppm C and O under the above conditions is reduced to less than 5% at room temperature to 1000°C.

Additional tensile tests of brazed Hastelloy X specimens show that the strength and ductility are strain rate sensitive with lower properties at slow strain rates.

6. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE

A total of 38 Ir-0.3% W PICS forming disks (lots TR 512-535) were shipped to MRC this quarter for the SIPS heat source. In addition, we shipped 38 disks previously rejected for flight hardware due to minor defects. Iridium foil was delivered to BCL for vent development studies.

We confirmed that the grain growth behavior of the HD production lots of iridium sheet was uniform and the same as the developmental DOP-4 sheets. The large grain size observed in the fuel impact tests after aging at LASL is not consistent with our data on sheet samples or samples from simulant FSA tests on the same lot of material. Additional tensile impact tests of

the HD production lots of the DOP-4 iridium alloy show that the tensile ductility is $19 \pm 4\%$ elongation for specimens annealed 19 hr at 1500°C . The number of grain boundaries across the thickness of these specimens ranged from 7-12. The TR lot of recycled iridium (30 ppm Th dopant addition) has an impact elongation and grain size on the lower end of these ranges.

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 Iridium Alloys — C. T. Liu and H. Inouye

1.2.1.1 Alloy Preparation and Fabrication. — Previous studies indicate that doping with a high level of thorium or a combination of thorium and aluminum significantly improves the high-velocity impact properties and grain stability of Ir-0.3% W alloys. Based on these results, we have prepared and characterized four additional doped alloys, designated as DOP-14-1, DOP-26, -27, and -28, as candidate alloys for improved MHW and general-purpose heat source applications. The starting material for these alloys was electron-beam melted, and dopants were added as arc-melted master alloys. The alloy composition and preparation are described below.

(1) DOP-14-1 — This is an Ir-0.3% W alloy doped with 200 ppm Th. This alloy was prepared for the purpose of further characterizing the metallurgical and mechanical properties of the DOP-14 alloy developed previously.¹ The alloy button was arc melted six times then drop cast into the full-size

*Progress on work performed under Activity No. KJ 06 01 01 0, 189a No. 01405.

ingot [$19 \times 19 \times 54$ mm ($3/4 \times 3/4 \times 2 \frac{1}{8}$ in.)]. The alloy stuck to the copper hearth several times during arc melting. However, the alloy button was acid etched after each melt to minimize contamination.

(2) DOP-27 — This alloy, which is a modified DOP-14 composition, was doped with 200 ppm Th and 50 ppm Al. The alloy button was arc melted six times then drop cast into the full-size ingot. The alloy stuck slightly to the copper hearth during arc melting.

(3) DOP-28 — On the live fuel compatibility test at LASL, tungsten has been found sometimes in the metallic crust at the vent ports.² It is possible that the tungsten may come from the Ir-0.3% W PICS, so we prepared the DOP-28 alloy, which is pure iridium doped with 200 ppm Th, for evaluation. The alloy button was prepared by electron-beam melting and drop casting into the full-size ingot.

(4) DOP-26 — This Ir-0.3% W alloy, which may be considered as an "upgraded" DOP-4, was doped with 60 ppm Th and 50 ppm Al. The alloy button was arc melted six times then drop cast into the full-size ingot. The alloy stuck to the copper hearth several times during arc melting.

The four ingots were separately canned in a molybdenum frame and fabricated into 0.074-cm- (0.030-in.-) thick sheets using the WGD production rolling schedule.³ All the sheets had good quality. The dopant concentration and trace impurities in these alloys were determined by SSMS analysis; the results are shown in Table 1.1. The tungsten content was determined from iridium-tungsten standards, and Al, Th, Ni, Fe, Ta, Pt, and Rh were measured by comparison with the tungsten in the samples. Although the alloys stuck to the copper hearth during arc melting, DOP-14-1 contains only 50 ppm Cu and the rest of the alloys contain less than 10 ppm. The acid etching after each melt appears to have minimized the contamination. The aluminum and thorium contents in DOP-14-1, DOP-26, and -27 are quite close to their nominal amount, indicating that the dopant concentration can be well controlled by arc melting. The reported thorium content in electron-beam-melted DOP-28 is much lower than the nominal content, probably due to lack of the reference standard (tungsten) in this alloy. The Fe, Cr, and Ni contents are quite low at a level ≤ 10 ppm in these alloys.

Table 1.1. Spark Source Mass Spectroscopic
(SSMS) Analysis of Alloy Specimens
DOP-14-1, DOP-26, -27, and -28

Element	Concentration by Weight (ppm)			
	DOP-14-1	DOP-26	DOP-27	DOP-28
Ag	1	—	—	3
Al	1	40	40	1
Ca	<0.1	<0.1	<0.1	<0.1
Cr	0.1	3	0.5	0.3
Cu	50	1	1	5
Fe	1	10	10	1
Hf	—	<1	—	—
K	—	30	30	—
Mo	3	3	3	3
Na	<0.1	0.1	≤0.1	<0.1
Ni	1	5	1	1
P	<0.1	<0.3	<0.3	<0.1
Pd	<0.1	<0.1	<0.1	<0.1
Pt	50	50	100	20
Rh	10	15	15	3
Ru	<0.1	0.5	60	<0.1
S	0.3	1	1	0.5
Si	<1	1	1	1
Ta	20	≤10	≤10	20
Th	200	50	150	115
Ti	<1	—	—	<1
W	2000	2200	2200	20

1.2.1.2 Tensile Properties. — The tensile properties of these four alloys were determined at 650°C after a standard heat treatment of 1 hr at 1500°C. The results are presented in Table 1.2 and are compared with those of undoped alloy WG. The yield and tensile strengths of doped alloys appear to increase with the total amount of aluminum and thorium, and is higher than that of undoped WG alloy. WG specimens had 30.1% elongation and the doped alloys had 35.4 to 39.5% at 650°C. Most significantly, all four doped alloys showed mainly transgranular fracture while the undoped alloy exhibited mainly grain-boundary separation at 650°C. It was noted that necking was visible on the fracture section of these doped alloys at 650°C.

Table 1.2. Tensile Properties^a of Undoped and Doped Iridium and Ir-0.3% W Sheet Specimens^b Tested at 650°C

Alloy Number	Nominal Dopant Concentration (ppm)	Strength, MPa (psi)		Elongation (%)	Fracture Mode ^c
		Yield	Tensile		
WG ^d	None	51.0 (7,400)	487.8 (70,800)	30.1	Mainly GBS
DOP-26 ^e	60 Th, 50 Al	75.8 (11,000)	529.8 (76,900)	35.4	Mainly TF
DOP-28 ^f	200 Th	95.1 (13,800)	513.3 (74,500)	39.3	Mainly TF
DOP-14 ^e	200 Th	93.7 (13,600)	556.0 (80,700)	37.4	Mainly TF
DOP-27 ^e	200 Th, 50 Al	97.2 (14,100)	564.3 (81,900)	37.5	Mainly TF

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

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

^cGBS = grain-boundary separation; TF = transgranular fracture.

^dUndoped Ir-0.3% W alloy prepared by electron-beam melting.

^eDoped Ir-0.3% W alloy prepared by arc melting.

^fIridium doped with 200 ppm Th, prepared by electron-beam melting.

1.2.1.3 Impact Properties. — Sheet specimens of DOP-14-1, DOP-26, -27, and -28 were heat treated 19 hr at 1500°C then impact tested at 1350°C at a velocity of 85 m/sec (280 fps). The impact results are presented in Table 1.3 and are compared with undoped WG. All doped alloys had more than 34% elongation and 85% reduction of area, and their fracture mode was mainly ductile rupture. In comparison, undoped alloy (WG) had 10.7% elongation and 23% reduction of area, and its fracture mode was grain-boundary separation. Also, impact properties of the new doped alloys are much better than those of DOP-4-type production material (reported in section 7.4 of this report).

The grain size of these impact specimens was measured with their results shown in Table 1.3. Figure 1.1 shows the grain structure and the precipitates (probably Ir₅Th) of these alloys. The DOP-26 specimen containing 60 ppm Th and 50 ppm Al had the coarsest grain structure after being annealed 19 hr at 1500°C. DOP-27, -28, and DOP-14-1 specimens all contain 200 ppm Th; however, DOP-28, prepared from electron-beam melting without the 0.3% W addition had 16.8 grains across the thickness while

Table 1.3. Tensile Impact Properties of Doped and Undoped Iridium and Ir-0.3% W Sheet Specimens Annealed 19 hr at 1500°C and Tested at 85 m/sec (280 fps) and 1350°C

Alloy Number	Grain Size ^b	Elongation (%)	Reduction of Area (%)	Fracture Mode ^a
WG-202 ^d	5.8	10.7 ^c	23 ^c	Mainly GBS
DOP-26 ^e	13.7	38.0	94	DR
-28 ^f	16.8	34.5	85	DR
-14-1 ^e	19.6	37.8	92	DR
-27 ^e	21.1	41.9	95	DR

^aDR = ductile rupture; GBS = grain-boundary separation.

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

^cAverage of two tests.

^dUndoped Ir-0.3% W alloy.

^eDoped Ir-0.3% W alloy.

^fDoped iridium.

Fig. 1.1. Microstructures of (a) DOP-26, (b) DOP-28, (c) DOP-14-1, and (d) DOP-27 Specimens Annealed 19 hr at 1500°C. 100×.

DOP-27, containing an additional 50 ppm Al (prepared by arc melting) had 21.1 grains across the specimen after the same heat treatment.

Tensile impact properties of the doped alloys DOP-4-11, DOP-23, -24, and -25 were reported previously⁴ after being annealed 1 and 19 hr at 1500°C. Table 1.4 summarizes the tensile impact properties of these alloys after a severe heat treatment of 1 hr at 1500°C plus 1 hr at 1800°C plus 1 hr at 1500°C. For comparison, the impact properties of undoped WG and thorium-doped WTh-1 are also shown in Table 1.4. The alloys DOP-24, -25, and DOP-4-11, doped with a combination of 30 ppm Th and 40-200 ppm Al, had better impact properties than those of DOP-23, doped with 40 ppm Al alone, and WTh-1, doped with 50 ppm Th alone. The DOP-24, -25, and DOP-4-11 specimens had about the same impact properties, indicating that doping with more than 40 ppm Al had no significant effect on improving the impact properties of Ir-0.3% W alloy under this severe heat treatment condition. The grain size of these impact specimens was measured with their results also listed in Table 1.4.

Table 1.4. Tensile Impact Properties of Doped and Undoped Ir-0.3% W Alloy Sheets Tested at 85 m/sec (280 fps) and 1350°C after Heat Treatment of 1 hr at 1500°C plus 1 hr at 1800°C plus 1 hr at 1500°C

Alloy Number	Dopant Concentration ^a (ppm)	Grain Size ^b	Elongation (%)	Reduction of Area (%)	Fracture Mode ^c
WG	None	2.4 ^c	3.4 ^d	6 ^d	Completely GBS
DOP-23	40 Al	2.3	4.7	11	Mainly GBS
WTh-1	50 Th	4.2	7.2	17	Mainly GBS
DOP-24	30 Th, 100 Al	4.4	9.8	20	Mainly GBS
DOP-25	30 Th, 200 Al	4.9	10.8	21	Mainly GBS
DOP-4-11 ^e	DOP-4 ^e plus 40 Al	3.8	10.3	17	Mainly GBS

^aNominal concentration.

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

^cGBS = grain-boundary separation.

^dAn average of two tests.

^eAlloy prepared from skulls and heads of production material HD-460 and -461 with 40 ppm Al added through arc melting.

1.2.2 Weldability of Improved Iridium Alloys — G. M. Goodwin

Studies are underway to characterize the weldability of iridium alloys with controlled additions of thorium and aluminum. Table 1.5 summarizes the results of preliminary weldability screening tests. Welds were performed on small coupons of available material and, thus, cannot be expected to accurately predict weldability response of structural welds in typical hardware. Trends, however, should be significant, and alloys showing poor response on these preliminary tests clearly need more detailed weldability characterization. Gas-tungsten arc (GTA) welds were made in a helium atmosphere chamber with lateral oscillation, closely approximating conditions utilized by Mound Laboratory for fueling of hemispheres.⁵

The results of Table 1.5 show a clear dependence of cracking tendency on thorium level. All welds containing thorium levels of 100 ppm or more cracked, while those containing less did not. Aluminum in the amounts added does not appear to influence weldability. No difference in response was noted between electron-beam and GTA welds.

Metallographic examination of a number of weld specimens showed an increase in the amount of second phase particles in the fusion zone as a function of thorium level as compared to previously reported microstructures for the base metal.⁶ Figure 1.2 shows the weld structure typical of EB- and GTA-weld low-thorium alloys. Although the GTA weld shows a generally coarser weld structure, no cracking is observed in either. By comparison, Figure 1.3 shows the structure typical of the higher-thorium-containing alloys. Note the general increase in the amount of second phase particles as the thorium level increases. The particles appear evenly distributed between grain boundaries and grain interiors. The hot cracking follows the weld centerline and shows little, if any, preference between transgranular and intergranular modes. Scanning electron microscopy (SEM) is being performed on fracture surfaces to further characterize this hot cracking behavior. Additional welding development is underway to determine the limiting thorium concentration and optimum weld parameters to prevent hot cracking.

Table 1.5. Weldability Screening of Doped Iridium Alloys

Alloy	Dopants (ppm)	Weld Process ^a	Results
WTh-1	50 Th	EB	no cracking
DOP-4-11D	30 Th	EB	no cracking
DOP-23D	40 Al	EB, GTA	no cracking
DOP-24D	30 Th, 100 Al	EB, GTA	no cracking
DOP-25D	30 Th, 200 Al	EB, GTA	no cracking
DOP-26C	60 Th, 60 Al	EB, GTA	no cracking
DOP-22	100 Th	EB, GTA	cracking
DOP-14	200 Th	EB, GTA	cracking
DOP-27C	200 Th, 50 Al	EB, GTA	cracking
DOP-28B	200 Th	EB, GTA	cracking
DOP-21	500 Th	EB, GTA	cracking

^aWelds in 0.64-mm (0.025-in.) sheet; EB = electron beam, GTA = gas tungsten-arc welds in helium atmosphere chamber with lateral torch oscillation.

^bAll cracked specimens showed centerline cracking of entire weld bead length.

(a)

(b)

Fig. 1.2. Typical Welds in Doped Iridium Alloy, DOP-25, Containing 30 ppm Th and 200 ppm Al. Note absence of second phase in (a) electron-beam weld and (b) gas tungsten-arc weld. 50 $\times$.

Fig. 1.3. Typical Welds in Doped Iridium Alloys Containing Various Thorium Content Levels. Note increase in second phase particles with increase in thorium content: (a) DOP-22 (100 ppm Th); (b) DOP-14 (200 ppm Th); and (c) DOP-21 (500 ppm Th). 100 $\times$.

1.2.3 Selection of An Improved Iridium Alloy for 1981-1983 Flight System -
C. T. Liu, H. Inouye, and A. C. Schaffhauser

To meet the development and qualification schedules for an improved iridium alloy for the improved Multihundred Watt (MHW) and General Purpose Heat Source (GPHS) applications, we must select at this time an alloy for more detailed characterization. This alloy must be technologically ready by the second quarter of FY 1978 (January through March) in order to meet the MHW flight mission in FY 1981 and GPHS design and qualification program for a FY-1983 mission. Table 1.6 compares the impact properties of the promising alloys developed recently with the undoped alloy WG and the current HD-type production material (DOP-4 composition, EB melted). Clearly, the impact properties of DOP-26 (doped with 60 ppm Th and 50 ppm Al) and DOP-14 (doped with 200 ppm Th) are much superior to the current production material, HD, when impacted at 85 m/sec (280 fps) at 1350°C. The impact properties of DOP-14 are even better than those of DOP-26 after a severe heat treatment at 1800°C; however, our initial welding studies show that alloys containing 100 ppm Th or more are subject to hot cracking due to extensive precipitation of second-phase particles (probably ThIr_5) in the fusion zone. Unless the weldability of the DOP-14 alloy can be improved in time, the best alloy for near-term impact property improvement appears to be the DOP-26 alloy, containing a combination of thorium and aluminum dopants. The DOP-26 alloy also has excellent ductility at lower impact temperatures (see section 2.2.1 of this report). For example, DOP-26 had 30.4% impact elongation while HD had only 14.4% elongation when impacted at 1050°C and 85 m/sec (280 fps). To retain the beneficial aluminum addition, it is necessary to arc melt the DOP-26 alloy, and the reproducibility of this alloy must be demonstrated under production conditions. Further qualification of this alloy will involve fuel compatibility tests at LASL, further demonstration of its weldability at ORNL and Mound Laboratory, and Fuel Sphere Assembly (FSA) impact testing at LASL.

We are continuing welding development on the DOP-14 alloy and will supply samples of this alloy for fuel compatibility testing at LASL. We recommend that the DOP-4-11 alloy (arc-melted 30 ppm Th, 40 ppm Al) be considered as a back-up improved alloy at this time.

Table 1.6. Tensile Impact Properties of Selected Doped Ir-0.3% W Alloys
Tested at 85 m/sec (280 fps) at 1350°C

Alloy	Nominal Dopant Concentration (ppm)	Melting Method ^a	Impact Results after Indicated Vacuum Heat Treatment					
			19 hr at 1500°C			1 hr at 1800°C ^b		
			Grain Size ^c	Elongation (%)	Fracture Mode ^d	Grain Size ^c	Elongation (%)	Fracture Mode ^d
WG ^e	None	EB	5.8	10.7	GBS	2.4	2.2	GBS
HD ^{e,f}	30 Th, 40 Al ^g	EB	8-11	15-23	TF + GBS	—	—	—
DOP-4-11 ^f	30 Th, 40 Al ^h	Arc	10.2	30.1	DR	3.8	10.3	GBS
DOP-25	30 Th, 200 Al ^h	Arc	12.8	32.0	DR	4.9	10.8	GBS
DOP-26	60 Th, 50 Al	Arc	13.7	38.0	DR	5.1	13.5	GBS
DOP-14-1	200 Th ^h	Arc	19.6	37.8	DR	12.0	25.2	TF + DR

^aEB = electron-beam melted and drop cast; arc = nonconsumable arc melted.

^bActual heat treatment: 1 hr at 1500°C + 1 hr at 1800°C + 1 hr at 1500°C.

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

^dDR = ductile rupture; TF = transgranular fracture; and GBS = grain-boundary separation.

^eMHW PICS production material.

^fImpact properties are not significantly affected by additions of 80 ppm Fe, 16 ppm Ni, and 75 ppm Th.

^gDopants added as elemental powders to iridium powder prior to melting.

^hDopants added as master alloy by arc melting to electron-beam-melted Ir-0.3% W starting stock.

1.3 Pt-Rh-W Alloys

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

We are continuing scale-up and fabrication development of the Pt-3008 (Pt-30% Rh-8% W) alloy for isotope fuel containment and conversion system structural applications. Our objective is to develop and evaluate melting and fabrication processes leading to a process and material specification for commercial procurement of Pt-3008. We are also utilizing the material fabricated in this task to supply Pt-3008 sheet for testing and development in other program tasks. Progress during this reporting period includes completion of sheet production from E2X (second consumable vacuum-arc-melted ingot)⁷ and IMPI-1 (an induction-melted and poured slab casting).⁸ Table 1.7 indicates recipients and anticipated uses of the fabricated sheets. It also shows, for each starting rolling billet, the number, total weight, and average thickness of sheets from the fabrication. The identity of a rolling billet in Table 1.7 prefixes the identification of each sheet that was fabricated from that billet. Fabrication history of the sheets is as follows.

As reported previously,^{7,8} the rolling billets cut from E2X extruded bars, as well as the cropped IMPI-1 slab casting, were hot rolled at 1200°C and, in most cases, then warm rolled at either 1000 and/or 1050°C. Table 1.8 characterizes the rolling billets at this stage of fabrication in terms of last elevated rolling temperature, thickness, and heat-treating temperature in preparation for cold rolling. Sequential cold rolling and 1100°C-vacuum-heat-treating operations, if required, then produced the sheet thickness in Table 1.7. For example, sheets from E2X-1A, -2A, and -1B were cold rolled 50% in thickness and supplied without final heat treatment. The concluding history of sheets from IMPI-1, however, included cold rolling to a reduction in thickness of 42%, heat treating 1 hr at 1100°C, further cold rolling of 50% to final thickness, and then a final stress-relieving heat treatment at 950°C in vacuum. All remaining sheets from E2X-7B, -6B, -5B, -4B, -3B, and -2B billets in Table 1.8 were processed similarly to IMPI-1.

Fabrication of the nine E2X and one IMPI rolling billets yielded, respectively, 71.8 and 79.6 wt % of their total starting weights as completed sheets. Trimming operations, which were sometimes required at the

Table 1.7. Characterization and Distribution of Pt-3008 Sheets
from E2X and IMPI-1 Heats

Billet Identity	Sheet Production				
	Number	Weight ^a (g)	Thickness mm(in.)	Recipient	Anticipated Use
E2X-1A	4	393	0.4826(0.019)	ORNL ^b	compatibility tests
-2A	2	374	0.4826(0.019)	ORNL	compatibility tests
-7B	1	329	0.254(0.010)	BCL ^c	vent fabrication
	1	356	0.2794(0.011)	MBC ^d	bellows fabrication
-6B	2	759	0.254(0.010)	MBC	bellows fabrication
-5B	2	624	0.254(0.010)	BCL	vent fabrication
-4B	1	273	0.127(0.005)	BCL	vent fabrication
	1	199	0.254(0.010)	BCL	vent fabrication
-3B	2	275	0.127(0.005)	BCL	vent fabrication
	2	41	0.254(0.010)	ORNL	weld test
	1	392	0.2794(0.011)	MBC	bellows fabrication
-2B	1	648	0.2794(0.011)	MBC	bellows fabrication
	1	39	0.2794(0.011)	ORNL	ultrasonic standard
-1B	1	<u>180</u>	0.4826(0.019)	ORNL	compatibility tests
TOTAL E2X WEIGHT		4882			
IMPI-1	1	918	0.2794(0.011)	MRC	bellows fabrication
	3	2856	0.2794(0.011)	ORNL	weld test; excess
	1	<u>146</u>	0.508(0.020)	ORNL	mechanical property tests
TOTAL IMPI WEIGHT		3920			

^aTo obtain pounds, multiply by 0.002.

^bOak Ridge National Laboratory, Tennessee.

^cBattelle Columbus Laboratories, Ohio.

^dMetal Bellows Corporation, California.

Table 1.8. Characterization of Billets before Cold Rolling

Billet Identity	Last Rolling Temperature ^a (°C)	Subsequent Heat-Treating Temperature ^a (°C)	Thickness [mm(in.)]	Remarks
E2X-1A	1000	1000	1.016(0.040)	nose of incomplete extrusion
-2A	1000	1000	1.016(0.040)	next section
-7B	1150	1150	1.524(0.060)	nose of completed extrusion
-6B	1200	1200	1.524(0.060)	next section
-5B	1050	1100	2.7178(0.107)	next section
-4B	1200	1200	1.27(0.050)	next section
-3B	1000	1100	2.159(0.085)	next section
-2B	1000	1000	1.016(0.040)	tail of completed extrusion
IMPI-1	1050	1100	1.524(0.060)	

^aTo obtain °F, multiply by 1.8 and add 32.

initiation and/or continuation of cold rolling, accounts for about 20 wt % of the reduced material yield from an individual billet. Table 1.9 presents this result. That is, the material which was trimmed from E2X-1A, -2A, -6B, -3B, -2B, -1B, and IMPI-1 billets to obtain rectangular-shape pieces for cold rolling averaged about 20 wt %. The relative starting weight of E2X-1B, which was only about one quarter of the others, magnifies the material lost value from this billet.

Attempts to initiate cold rolling of E2X-7B, -5B, and -4B billets at a thickness of about 2.6 mm (0.10 in.), however, lowered the overall yield of material from about 80 to the 71.8 wt %. As indicated in Table 1.9, two billets delaminated due to inhomogeneous deformation from the small cold reduction passes (E2X-5B and -7B) and one formed a severe edge crack (E2X-4B) when it was flattened to facilitate holding in a subsequent operation. These billets were repaired, but not without the significant loss of material. Their intermediate fabrication steps deviated from that of the other E2X billets. Fortuitously, the total delamination of E2X-5B occurred quite near to and uniformly along the midplane of the billet.

Table 1.9. Yield of Fabricated Sheet from
E2X and IMPI-1 Rolling Billets

Billet Identity	Yield (wt %)	Remarks
E2X-1A	83	
-2A	79	
-7B	72	Limited delamination after initial 4% cold reduction
-6B	82	
-5B	64	Delamination halved billet after initial 6% cold reduction
-4B	48	Curved severely during warm roll- ing and initial 3% cold reduction; attempts to flatten formed long curving crack
-3B	80	
-2B	79	
-1B	74	Smallest billet; relative losses magnified
IMPI-1	80	

The exposed surfaces therefore required only minor amounts of grinding to prepare them for cold rolling from a thickness of about 1.2 mm (0.05 in.). Unfortunately, the long curving crack in E2X-4B was located near the center of the billet. After removal of the crack from E2X-4B and also both delaminated ends from E2X-7B, hot rolling, followed by heat treating at the rolling temperature, reduced these billets to the thickness shown in Table 1.8.

1.3.2 Characterization of Pt-3008 Scale-Up Heats — J. R. Keiser

The objective of this task is to characterize and determine the factors controlling the properties of Pt-3008, particularly the large change in ductility that generally occurs near room temperature and the ductility minimum that occurs around 1100°C. In previous work, it has been found that the properties are affected by many factors including impurity concentrations and annealing and aging treatments. These prior investigations

have been carried out over an extended time and, of necessity, on a variety of heats. In order to better relate some of these results, we are repeating the more significant, but are using one heat for all experiments. For this heat (the second scale-up heat, E2X) we will also obtain a complete chemical analysis, a measure of the grain size of certain specimens, and an Auger examination of the fracture surface of specimens displaying certain properties. From this we hope to advance our understanding of what part bulk impurities, the grain size, and/or the chemistry of the fracture surface play in controlling the mechanical properties of Pt-3008.

Results we have obtained include a complete chemical analysis of heat E2X and the tensile strength and elongation as a function of test temperature for specimens annealed and aged several ways. The results of the analysis are given in Table 1.10. Care should be exercised in evaluating this data because of previous experience in which tests of pieces from the same heat yielded greatly different results. Whether this is due to real compositional variations in the alloys or to lack of reproducibility of the analysis technique is not known.

The results of tensile tests of Pt-3008 specimens annealed 1 hr at 1200°C in vacuum are shown in Fig. 1.4. The low ductility at room temperature and the ductility minimum around 1100°C are apparent. Specimens which had received an aging treatment of 1000 hr at 1100°C in a static vacuum following the 1-hr-at-1200°C standard anneal showed better ductility at room temperature and 1100°C but showed a large decrease in strength and ductility at 1316°C as can be seen in Fig. 1.5. This reduction in mechanical properties may be due to silicon contamination from the quartz encapsulation tube and this will possibly be investigated using Auger analysis of fracture surfaces. A 200-hr-at-1100°C aging treatment in hydrogen following the standard anneal had a significant effect only on the properties around the 1100°C ductility minimum as is shown in Fig. 1.6. Another set of specimens has been aged 1000 hr at 1100°C in a dynamic vacuum and is currently being tested.

Metallography, scanning electron microscopy, and Auger spectroscopy are being used when appropriate to examine the fracture surfaces of these specimens in an effort to explain the observed behavior. We are theorizing that the disappearance of the ductility minimum is a result of removal of an interstitial element, possibly B, O, or C, by the aging treatments. We have not currently obtained any results to support this theory.

Table 1.10. Chemical Analysis
Results for Pt-3008 Second
Scale-Up Heat^a

Element	Sample 1 (ppm)	Sample 2 (ppm)
Ag	<0.1	<0.1
Al	3	3
B	1	1
C	33	29
Ca	0.3	0.1
Cl	1	1
Co	10	10
Cr	30	30
Cu	50	20
Fe	150	150
H	2	4
Hf	0.5	0.5
Ir	≤30	≤30
Mg	0.5	<0.5
Mn	3	3
Mo	100	50
N	0.96	0.80
Na	<0.1	<0.1
Ni	50	50
O	7	15
Os	10	10
P	1	1
Pd	5	5
Re	3	3
Ru	5	5
S	1	1
Si	3	10
Sn	1	1
Ta	10	10
Th	20	5
V	3	1
Zn	<0.1	<0.1
Zr	10	10

^aSample 1 had 26.0% Rh and 7.0% W; sample 2 had 29.0% Rh and 7.2% W.

ORNL-DWG 76-19938

Fig. 1.4. Elongation vs Temperature of Pt-3008 Second Scale-Up Heat (Annealed 1 hr at 1200°C).

Fig. 1.5. Elongation vs Temperature of Pt-3008 Second Scale-Up Heat After a 1-hr-at-1200°C Anneal and After Aging of 1000 hr at 1100°C in Static Vacuum.

ORNL-DWG 77-3818

Fig. 1.6. Elongation vs Temperature of Pt-3008 Second Scale-Up Heat After a 1-hr-at-1200°C Anneal and After Aging 200 hr at 1100°C in H₂.

1.3.3 Welding of Pt-3008 Alloy — G. M. Goodwin and J. D. Hudson

Testing of weldments and base metal from the first scale-up heat (E1) is continuing. Base metal data from recent tests are summarized in Table 1.11, with previously reported tests for comparison. Specimens E1-22 through -24 were machined from sheet to determine the effects, if any, of the normal punching technique for specimen preparation. As is seen from the table, the data show no significant consistent effect of specimen preparation technique. Yield strength appears to be somewhat lower for machined specimens, but this property is subject to significant scatter. Ductility values are within the anticipated ranges. Additional machined specimens are being tested in the recrystallized condition to determine if there is any improvement in the ductility and scatter in data previously observed.

Table 1.11. Tensile Properties of Pt-3008^a

Specimen Designation	Specimen Type ^b	Test Temperature (°C)	Strength, MPa(ksi)		Total Elongation (%)
			0.2% Offset	Ultimate	
E1-22	B-M	20	910.1(132.0)	1420.4(206.0)	14.7
-23	B-M	200	772.2(112.0)	1332.1(193.2)	13.0
-24	B-M	400	689.5(100.0)	1180.4(171.2)	10.0
-1	B	20	1149.4(166.7)	1393.4(202.1)	12.0
-2	B	20	1149.4(166.7)	1393.5(202.1)	12.0
-16	B	400	—	1221.0(177.0)	11.8
-18	B	400	1149.1(166.7)	1307.2(189.6)	12.0
-19	B	760	948.1(137.5)	999.8(145.0)	8.5

^aSheet, 0.56-mm- (0.022-in.-) thick, from heat E1, stress relieved 1 hr at 850°C.

^bB = base metal specimens punched by usual technique; B-M = base metal specimens machined.

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 Rhodium-Ruthenium Alloys

A series of rhodium-ruthenium alloys with the compositions listed in Table 1.12 have been or will be evaluated. The alloys containing less than approximately 50 wt % Ru should be fcc and those alloys with greater than 60 wt % Ru are expected to be cph.⁹ To date, the alloys having the fcc structure (45 wt % Ru or less) appear to be fabricable while the cph alloys are not.

Table 1.13 lists the tensile properties of rhodium sheet prepared from an arc-melted and drop-cast ingot. The strength of this heat is considerably higher and the ductility lower than those reported for rod specimens¹⁰ (also listed in the table).

Table 1.12. Composition of
Rhodium-Ruthenium Alloys

Alloy Designation	Ruthenium Content (wt %)	Status
44-28	0	rolled to sheet
-32	9.8	ingot cast
-09	20.0	rolled to sheet
-29	34.6	rolled to plate
-30	44.6	rolled to plate
-31	64.5	ingot cast
-08	80	not fabricable
-19, -20, -21	100	not fabricable

Table 1.13. Tensile Properties of Rhodium in Vacuum^a

Strength, MPa(ksi)		Elongation (%)	Reduction of Area (%)
Ultimate	0.2% Offset Yield		
<u>25°C</u>			
543.6(78.9)	454.0(65.9)	2.3	1.3
<u>500°C</u>			
526.4(76.4)	370.7(53.8)	11.2	7.8
<u>750°C</u>			
406.5(59.0)	340.4(49.4)	15.2	15.7
335.5(48.7)	106.1(15.4)	29	85 (Ref. 10)
<u>1000°C</u>			
273.5(39.7)	226.0(32.8)	13.3	17.4
117.1(17.0)	47.5(6.9)	61	100 (Ref. 10)

^aHeat 44-28 rolled to sheet from arc-drop cast ingot. Gage dimensions: 12.7 × 3.2 × 0.4 mm (0.5 × 0.125 × 0.015 in.). Annealed 1 hr at 800°C. Strain rate: 0.2 min⁻¹.

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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 extensively 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 Effect of Test Temperature on Impact Properties of Ir-0.3% W Alloys — C. T. Liu

Tensile specimens of DOP-4 (30 ppm Th, 40 ppm Al), DOP-14 (200 ppm Th), and DOP-26 (60 ppm Th, 50 ppm Al) alloys were heat treated 1 and 19 hr at 1500°C, then impact tested at 1050°C and 85 m/sec (280 fps). The results are presented in Table 2.1 together with those of undoped WG specimens. Compared with the impact data obtained at 1350°C, all the alloys had lower ductility at 1050°C. However, the DOP-26 alloy remains ductile at 1050°C and has the best impact properties among the alloys. The impact ductility decreases with an increase in annealing time from 1 to 19 hr at 1500°C. This suggests that the impact properties of the alloys at 1050°C are also dependent on the heat treatment or grain size.

*Progress on work performed under Activity No. KJ 06 01 01 0, 189a No. 01406.

Table 2.1. Tensile Impact Properties of Doped and Undoped Ir-0.3% W Sheet Specimens Tested at 1050°C and 85 m/sec (280 fps)

Alloy Number	Nominal Dopant Concentration (ppm)	Grain Size ^a	Elongation (%)	Reduction of Area (%)	Fracture Mode ^b
<u>Annealed 1 hr at 1500°C</u>					
WG	None	10.7	5.4	13	Mainly GBS
DOP-4	30 Th, 40 Al ^c	19.2	14.4 ^d	35 ^d	TF and GBS ^d
DOP-26	60 Th, 50 Al ^e	—	30.4	55	DR and TF
DOP-14	200 Th ^e	25.3	29.2	69	DR and TF
<u>Annealed 19 hr at 1500°C</u>					
WG	None	5.8	—	—	—
DOP-4	30 Th, 40 Al ^c	10.2	9.3	18	Mainly GBS
DOP-26	60 Th, 50 Al ^e	13.7	20.1	40	TF (Ma) and GBS (Mi)
DOP-14	200 Th ^e	19.6	18.2	31	TF and GBS

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

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

^cDopants listed plus 80 ppm Fe, 16 ppm Ni, 75 ppm Rh added as elemental powders to iridium powder prior to electron-beam melting.

^dAn average of two tests.

^eDopants added by arc melting to electron-beam-melted Ir-0.3% W starting stock.

2.2.2 Effect of Long-Term Heat Treatment at 1330°C on Impact Properties of Ir-0.3% W Alloys — C. T. Liu and D. E. Harasyn

To study the effects of long-term heat treatment at the MHW clad temperature on the metallurgical and mechanical properties of Ir-0.3% W alloys, DOP-4, -14, and WG specimens were annealed at 1330°C for different periods then impacted at 1350°C and 85 m/sec (280 fps). The impact results obtained for specimens annealed up to 6000 hr are summarized in Table 2.2 and are plotted as a function of annealing time in Fig. 2.1. The grain size of the impact specimens was also measured and shown in Table 2.2. The impact ductility of the three alloys decreases with increasing annealing time; this decrease is apparently due to grain growth as indicated in Table 2.2. The DOP-14 alloy had 36.4% impact elongation while DOP-4 is estimated to have approximately 17% and WG approximately 5% after a 5000-hr anneal. Thus, DOP-14 is much more resistant to degradation due to long-term heat treatment at 1330°C.

2.2.3 Impact Properties of Ir-0.3% W Alloys Under Abnormal Conditions — C. T. Liu

Under emergency cooling conditions, the fuel-clad temperature in the Brayton Isotope Power System (BIPS) may reach as high as 1660°C ($\sim$ 3075°F) for a transition period of 60 hr. Since the impact properties of iridium alloys are sensitive to grain size, we are characterizing the impact behavior of thorium-doped Ir-0.3% W alloys at this severe condition.

Sheet specimens of DOP-21 (500 ppm Th) and DOP-14 (200 ppm Th) were heat treated in vacuum for 60 hr at 1660°C then impacted at 1350°C and 85 m/sec (280 fps). The impact results are presented in Table 2.3 together with the grain size measured from the shoulder of the impact specimens. Both alloys had more than 23% impact elongation, suggesting that they would survive the MHW/FSA-type impact test under this severe heat treatment condition. DOP-21 had better impact ductility than DOP-14 because of its finer grain structure, as indicated in Table 2.3.

Table 2.2. Effect of Long-Term Anneal at 1330°C
on the Impact Properties of Doped and Undoped
Sheet Specimens Tested at 85 m/sec
(280 fps) and 1350°C

Specimen	Grain Size ^a	Impact Elongation (%)	Reduction of Area (%)	Fracture Mode ^b
<u>480-hr Anneal</u>				
WG ^c	8.3	8.4	24	Mainly GBS
HD ^d	15.2	26.5	90	DR
<u>1000-hr Anneal</u>				
WG ^c	7.3	8.6	20	Mainly GBS
HD ^d	11.0	27.2	88	DR
DOP-14 ^e	23.5	42.8	92	DR
<u>2000-hr Anneal</u>				
WG	4.7	7.8	19	Mainly GBS
HD	8.5	24.0	61	Mainly TF
<u>3000-hr Anneal</u>				
DOP-14	18.1	38.3	86	DR
<u>4000-hr Anneal</u>				
WG	4.4	6.4	14	Mainly GBS
HD	5.9	17.2	39	TF and GBS
<u>5000-hr Anneal</u>				
DOP-14	15.5	36.5	84	DR
<u>6000-hr Anneal</u>				
HD	5.2	16.2	34	TF and GBS

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

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

^cUndoped Ir-0.3% W alloy.

^dIr-0.3% W alloy doped with DOP-4 dopants.

^eIr-0.3% W alloy doped with 200 ppm Th.

Fig. 2.1. Effect of Long-Term Annealing at 1330°C on Impact Elongation of WG, DOP-4, and DOP-14 Specimens. All specimens were impacted at 85 m/sec (280 fps) and 1350°C.

Table 2.3. Tensile Impact Properties of DOP-14^a and DOP-21^b Alloys Heat Treated at 1660°C for 60 hr and Tested at 85 m/sec (280 fps) and 1350°C

Alloy	Grain Size ^c	Thorium Content (ppm)	Elongation (%)	Reduction of Area (%)	Fracture Mode ^d
DOP-14	8.6	200	23.3	53	Mainly TF
DOP-21	12.5	500	35.5	86	TF and DR

^aIr-0.3% W doped with 200 ppm Th.

^bIr-0.3% W doped with 500 ppm Th.

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

^dTF = transgranular fracture; DR = ductile rupture.

2.3 Environmental and Impurity Effects

2.3.1 Air Oxidation of Iridium Alloys — C. T. Liu

The Ir-0.3% W alloys doped with active elements such as thorium may show internal oxidation when exposed to air in the post-impact temperature range and, thus, seriously affect their ductility. To characterize the air oxidation behavior of Ir-0.3% W alloys, the tensile specimens of WG, DOP-4, -22, -14, and -21 (with their dopant concentrations listed in Table 2.4) are being exposed to still air at 670 and 870°C in tests scheduled to 5000 hr. At present, we have completed the 1000-hr exposure test. All the exposed specimens exhibited a thin, dark brown oxide coating on the surface. The specimens showed almost no weight change at 670°C but a weight loss at 870°C.

The air-oxidized specimens were then tested in tension at the exposure temperature. The tensile results are shown in Table 2.4. All exposed specimens have 35-40% elongation at 670°C and 42-52% elongation at 870°C, similar to the unexposed specimens.¹ Also, the tensile and yield strengths are the same as the unexposed specimens. Thus, air exposure to 1000 hr at 670 and 870°C does not degrade the tensile properties of doped and undoped Ir-0.3% W alloys.

Table 2.4. Tensile Properties^a of Undoped and Doped
Ir-0.3% W Sheet Specimens^b Exposed to
Air for 1000 hr at 670 and 870°C

Specimen	Dopant Concentration ^c (ppm)	Strength, MPa(psi)		Elongation (%)
		Yield	Tensile	
<u>Exposed and Tested at 670°C</u>				
WG	None	63.4(9,200)	471.3(68,400)	34.8
HD	DOP-4	81.3(11,800)	488.5(70,900)	34.9
DOP-22	100 Th	85.4(12,400)	503.0(73,000)	37.5
DOP-14	200 Th	99.9(14,500)	551.2(80,000)	38.5
DOP-21	500 Th	113.0(16,400)	567.1(82,300)	39.8
<u>Exposed and Tested at 870°C</u>				
WG	None	55.8(8,100)	334.9(18,600)	46.5
HD	DOP-4	64.8(9,400)	345.9(50,200)	51.8
DOP-22	100 Th	71.0(10,300)	380.3(55,200)	46.3
DOP-14	200 Th	86.8(12,600)	394.1(57,200)	43.6
DOP-21	500 Th	97.2(14,100)	401.7(58,300)	42.7

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

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

^cNominal concentration.

2.3.2 Pt-3008/Graphite Compatibility — J. R. Keiser

One proposed use of Pt-3008 is for encapsulation of $^{238}\text{PuO}_2$ isotope fuel. To be considered for this application, the alloy must demonstrate an ability to survive a launch pad accident in which a fuel capsule inside a graphite impact shell would fall onto a piece of burning solid propellant rocket fuel. Calculations have indicated the exposure conditions could be as severe as 1800°C for 5 min. The concern about using Pt-3008 for this application is the eutectic reaction which occurs between Pt-3008 and graphite below 1850°C. In order to determine the suitability of Pt-3008 for use in fuel encapsulation, we have taken pieces of graphite and the alloy, put them in contact and held them for 5 min at an elevated temperature. We have exposed specimens at temperatures of 1750, 1800, 1850, 1900, and 1950°C. Results of metallographic examinations, shown in Fig. 2.2, indicate significant but noncatastrophic reaction between Pt-3008 and

Fig. 2.2. Pt-3008 in Contact with POCO AXF-Q1 Graphite for 5 Min in Vacuum at (a) 1750, (b) 1800, (c) 1900, and (d) 1950°C.

graphite. For specimens tested below 1800°C, the microstructure shows spheres of graphite in the Pt-3008, while the microstructures of specimens tested at 1800°C and above show platelets of graphite indicative of localized eutectic reaction. Although there is a large amount of graphite in the higher-temperature specimens, Pt-3008 may be capable of providing the fuel containment required for this maximum credible accident condition.

A test has also been conducted to determine if a layer of iridium between the Pt-3008 and the graphite would prevent reaction, but, as can be seen in Fig. 2.3, the iridium did not significantly affect the results. Additional tests are being performed with Pt-3008 tensile specimens exposed to graphite at 1900°C then tested to determine the effect of exposure on mechanical properties.

Fig. 2.3. Pt-3008 with 5-Mil Ir-0.3% W Foil on Both Sides Contacting Same Graphite for 5 Min in Vacuum at 1900°C.

2.4 Microanalysis Studies

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

2.4.1 Analysis of Iridium Cladding from Impacted Simulant Fuel Sphere Assemblies

2.4.1.1 Introduction. — Preliminary examination of the iridium Post Impact Containment Shell (PICS) used in simulant fuel sphere impacts MHT-103 and -104 indicates that the impact performance of these PICS is significantly worse than that for PICS in MHT-90, -91, and -92.^{2,3} While MHT-90, -91, and -92 survived impact without breaches in the cladding, MHT-103 cracked along the closure weld and on the inside of the impact face (fingerprint cracking), and MHT-104 had a 19.05-mm- (3/4-in.-) diam piece broken out of the impact face due to fingerprint cracking. Because the failure mode during impact involved intergranular separation, a study of the grain boundary chemistries of these alloys, using Auger spectroscopy, has been initiated. Table 2.5 summarizes the samples examined up to this point. The results on samples from MHT-103 and -104 have been reported previously⁴ but are included here for comparison with results from MHT-90, -91, and -92.

Samples from MHT-90, -91, and -92 were prepared for analysis in a similar manner to samples from MHT-103 and 104; however, the following difference should be noted. Unlike samples from MHT-103 and -104, samples from MHT-90, -91, and -92 were baked out ($\sim 200^{\circ}\text{C}$) in the vacuum transfer lock prior to being placed in the AES-UHV system for Auger analysis. This procedure results in less oxygen contamination of the fracture surfaces, hence less attenuation of the thorium signaled as a function of time.⁵

2.4.1.2 Analysis and Results. — Samples 760135 and 760136 (MHT-91) were the first of the samples in Table 2.5 to be analyzed. Figure 2.4 shows the Auger spectrum from the center of the fracture surface of sample 760135. This spectrum shows carbon and oxygen peaks that are somewhat larger than normal, with thorium intensity at approximately normal levels for DOP-4-type alloys.

Table 2.5. Description of Samples Analyzed
Using Auger Electron Spectroscopy

Simulant Fuel Sphere Assembly	Fuel Cladding Heat	Performance in Simulant Fuel Impact Test	Auger Sample Number	Location
MHT-90	DOP-4-3	Microcracking in regions having dye penetrant indi- cations prior to impact	760157	Weld
	DOP-4-4		760158	Sheet
MHT-91	DOP-4-2	No cracks observed at 30× or by dye penetrant	760136	Weld
			760135	Sheet
MHT-92	DOP-4-4	No breach observed at 30×	760184	Weld
			760185	Sheet
MHT-103		1-in. crack along closure weld-fingerprint cracking on inside of impact face penetrating 50% of the wall thickness	760131	Weld
			760130	Sheet
MHT-104		3/4-in. piece broken out of impact face due to finger- print cracking	760133	Weld
			760132	Sheet

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Fig. 2.4. An Auger Spectrum from the Fresh Fracture Surface of Sample 760135 (MHT-91, w/o weld). This spectrum was taken while rastering the primary electron beam over a small area in the center of the fracture surface.

Figure 2.5 shows the Auger spectrum from near the inner edge* of the fracture surface and sample 760135. Large concentrations of S, P, C, and O are indicated in this spectrum and the 67-eV Th peak is almost undetectable.

In order to obtain a semi-quantitative comparison between spectra, a ratio of peak intensities can be used. The ratio of $I(P, 120 \text{ eV})$ to $I(\text{Ir}, 229 \text{ eV})$ in Fig. 2.5 is approximately 0.82. This number is approximately proportional to X_P (the atom fraction of P) divided by X_{Ir} (the atom fraction of Ir). Estimates of the ratio X_P/X_{Ir} can be obtained by multiplying the peak height ratio by $S_{\text{Ir}}^{\text{Ag}}/S_P^{\text{Ag}}$ where $S_{\text{Ir}}^{\text{Ag}}$ and S_P^{Ag} are the sensitivities of Ir and P with respect to a Ag standard. Approximate values of sensitivity factors for some of the Auger peaks observed in Fig. 2.5 are listed in Table 2.6. These sensitivity factors are estimated from standard spectra of pure elements and compounds in the *Handbook of Auger Electron Spectroscopy*.⁶ The use of sensitivity factors in the manner outlined above does not account for possible differences between the analyzer used to obtain the experimental data and the one used to generate the handbook spectra. Matrix, chemical, and distribution effects are also ignored using the procedure. Because of these complications and because relative differences in composition are of primary interest, the data from the Auger analysis of the samples in Table 2.5 will be presented as peak height ratios. Approximate atom fraction ratios could be estimated from the peak height ratios using the data in Table 2.6, however, those values should be used with considerable caution.

It should be pointed out that the measurement of $I(S, 152 \text{ eV})$ is hampered by the presence of the 154-eV Ir peak. In the case of Fig. 2.5, where $I(S, 152 \text{ eV})$ is much larger than $I(\text{Ir}, 154 \text{ eV})$, it is reasonable, for our purposes, to ignore the effect of the 154-eV Ir peak. For spectra where $I(S, 150 \text{ eV})$ is smaller, or not much larger than $I(\text{Ir}, 154 \text{ eV})$, interference between the two peaks makes measurement of $I(S, 152 \text{ eV})$ subject to considerable

*The "inner" edge of the fracture surface will refer to the edge of the fracture surface nearest the concave surface of the cladding. Using this definition, the inner edge of the fracture surface was nearest to the ThO_2 fuel simulant, and the outer edge of the fracture surface was nearest to the graphite impact shell (GIS).

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Fig. 2.5. An Auger Spectrum from the Fresh Fracture Surface of Sample 760135 (MHT-91, w/o weld). This spectrum was taken while the primary electron beam was located on a spot near the inner edge of the fracture surface.

Table 2.6. Sensitivity Factors^a
for Selected Auger Peaks

Element	Peak Energy (eV)	Sensitivity Factor (relative to 351 eV Ag)
Ir	54	0.30
Ir	154	0.047
Ir	229	0.049
Th	67	0.35
P	120	0.46
S	152	0.76
C	272	0.15
O	510	1.02 ^b

^aThese sensitivities are calculated from standard Auger spectra of elements and compounds found in the *Handbook of Auger Electron Spectroscopy*.⁶ Except where indicated, spectra taken with a 5-keV primary beam were used.

^bUsing standard spectrum for MgO with a 3-keV primary beam.

error. The values of $I(S, 152 \text{ eV})$ used in this study have been measured from the extreme positive deviation of the combined 152-eV S and 154-eV Ir peaks to the negative deviation of the 152-eV S peak.

The location of the spots where Auger spectra were taken from the fracture surface of sample 760135 are indicated in Fig. 2.6. The peak height ratios for elements indicated in these spectra are given in Table 2.7 along with the ratio:

$$x/t$$

where

x = the distance from the inner edge of the fracture surface to the spot analyzed, and

t = the distance from the inner edge to the outer edge of the fracture surface on the sample current image.

The data in Table 2.7 indicate contamination of the grain boundaries near the bottom edge of the fracture surface with P and S. Larger than normal concentrations of C and O are also indicated in all spectra, with

Fig. 2.6. A Sample Current Image of the Fracture Surface of Sample 760135, Showing the Spots Where Auger Spectra Were Taken.

Table 2.7. Summary of Auger Results for Sample 760135 (MHT-91, w/o Weld)

Chart	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 54 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{S}, 152 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 510 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761013 C-03 ^a	1	0	0	0.85	5.65	8.15	3.90
-04 ^a	2	0.04	0	0.82	4.66	2.92	1.80
-05 ^a	3	0.10	0.03	0.57	3.02	0.98	0.84
-06 ^a	4	0.17	0.08	0.56	3.02	0.67	1.02
-07 ^a	5	0.24	0.06	0	2.45	0.86	0.57
-08 ^a	6	0.30	0.06	0	0.90	0.62	0.40
-09 ^a	7	0.37	0.07	0	1.17	0.57	0.43
-10 ^a	8	0.44	0.04	0	0.50	0.40	0.29
-11 ^a	9	0.50	0.06	0	0.70	0.43	0.26
-12 ^a	10	0.58	0.02	0	0.54	0.46	0.25
-13 ^a	11	0.70	0.02	0	1.08	0.74	0.47
-14 ^a	12	0.84	0.04	0	1.35	0.81	0.34
-15 ^a	13	0.98	0	0	1.31	2.21	0.52
-16 ^a	14	0.91	0	0	1.03	0.79	0.42
A761020 C-03 ^b	3	0.10	0	0.04	0	0.33	0.21

^aSpectra taken on fresh fracture surface.

^bSputtered for 2 min. Sputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

the largest peak height ratios near the inner edge of the fracture surface. The Th levels indicated in Table 2.7 are somewhat lower than those normally observed in DOP-4-type Ir-0.3% W alloys; however, the presence of the O may be masking the Th peak. Inert ion sputtering experiments indicated that the P and S contamination was confined to within a few atom layers of the grain boundary.

The results of Auger analysis of sample 760136 are summarized in Table 2.8. The locations of the spots indicated in Table 2.8 are shown in Fig. 2.7. The inner edge of the fracture surface was contaminated with P, and slight indications of S were indicated at isolated locations near the outer and inner edges of the fracture surface. The level of Th was generally somewhat higher than for 760135; however, there was much variation from spot to spot.

Because of the high S, C, and O levels observed on sample 760135, samples from MHT-90 and -92 were acid cleaned prior to ultrasonic cleaning in alcohol in an effort to remove foreign material that might be embedded in pre-existing cracks.

The results of Auger analysis on samples from MHT-90 (760158 and 760157) are summarized in Tables 2.9 and 2.10, respectively. The locations of the spots where spectra are taken are shown in Figs. 2.8 and 2.9. Both of these samples had indications of P contamination at various locations near the inner edge of the fracture surface; however, the contamination did not appear to be continuous since other spots near the inner edge showed little or no evidence of P contamination. The Th-to-Ir peak height ratios in Tables 2.9 and 2.10 range from nearly twice the average values for DOP-4-type Ir-0.3% W down to very small values. Neither of the samples from MHT-90 had any indication of S contamination.

The data for samples from MHT-92 are summarized in Tables 2.11 and 2.12. The location of the spots where spectra were taken are indicated in Figs. 2.10 and 2.11. These results follow much the same pattern as those for MHT-91, with indications of P contamination near the inner edge of the fracture surface, and isolated spots of high S near the outer and inner edges.

2.4.1.3 Summary and Conclusions. — The data concerning the distribution of thorium and phosphorus on the fracture surfaces is summarized in

Table 2.8. Summary of Auger Results for Sample 760136 (MHT-91, with Weld)

Chart	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 54 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{S}, 152 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 510 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761014 C-07 ^a	1	0	0	1.64	0	0.59	0.62
-08 ^a	2	0.05	0.04	1.53	0	0.33	0.25
-09 ^a	3	0.12	0.08	0.96	0.29	0.58	0.40
-10 ^a	4	0.18	0.08	0.75	0.12	0.37	0.28
-11 ^a	5	0.23	0.06	0.48	0	0.37	0.33
-12 ^a	6	0.30	0.03	0	0	0.30	0.24
A761020 C-01 ^a	7	0.50	0.01	0	0	0.52	0
-02 ^a	8	0.90	0.14	0	0.09	0.58	0.37
-04 ^b	2	0.05	0	0	0	0.46	0.09

^aSpectra taken on fresh fracture surface.

^bSputtered for 2 min. Sputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

Fig. 2.7. A Sample Current Image of the Fracture Surface of Sample 760136, Showing the Spots Where Auger Spectra Were Taken.

Table 2.9. Summary of Auger Results for Sample 760158 (MHT-90, w/o Weld)

Chart	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 54 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 512 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761116 C-01 ^a	A	0.44	0.18	0	0	0
-03 ^a	1	0.29	0.13	0.07	0.12	0.12
-04 ^a	2	0.18	0.16	0.95	0.15	0.21
-05	3	0.11	0.06	1.08	0.13	0.15
-06	4	0.03	0.02	1.23	0.12	0.38
-07	5	0.98	0	0	0.58	0.95
-08	6	0.87	0.07	0	0.80	0.12
-09	7	0.71	b	0	0.29	0
-10	8	0.60	0.07	0	0.22	0.16
A761117 C-04 ^c	4	0.03	0	0	0.29	0.07
-05 ^c	2	0.18	0	0	0.32	0.08

^aSputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

^bOmitted from the spectrum was 67 eV Th.

^cSputtered for 2 min. Sputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

Table 2.10. Summary of Auger Results for Sample 760157 (MHT-90, with Weld)

Chart	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761116 C-11 ^a	1	0.51	0.15	0	0.08	0
-13 ^a	2	0.08	0.09	0.02	0.22	0.18
A761117 C-01 ^a	3	0.92	0.13	0.03	0.57	0.16
-02 ^a	4	0.08	0.03	0.02	0.45	0
-03 ^a	5	0.05	0.18	0.98	0.40	0.18
-06 ^b	5	0.05	0	0	0.26	0
-07 ^b	1	0.51	0	0	0.28	0

^aSpectra taken on fresh fracture surface.

^bSputtered for 2 min. Sputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

Fig. 2.8. A Sample Current Image of the Fracture Surface of Sample 760158, Showing the Spots Where Auger Spectra Were Taken.

Fig. 2.9. A Sample Current Image of the Fracture Surface of Sample 760157, Showing the Spots Where Auger Spectra Were Taken.

Table 2.11. Summary of Auger Results for Sample 760185 (MHT-92, w/o Weld)

Chart ^a	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 64 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{S}, 152 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 510 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761221 C-01	1	0.52	0.15	0.11	0	0.42	0.14
-03	2	0.03	0.01	0.86	0	0.86	0.24
-04	3	0.97	0.03	0.09	3.08	0.54	0.39
A761222 C-01	4	0.03	0.08	0.60	0	0.67	0.28
-02	5	0.03	0.05	0.29	2.67	1.24	0.51
-03	6	0.90	0.05	0.17	0	0.93	0.18
A761227 C-01	2	0.03	0.02	0.66	0	1.41	0.25

^aAll spectra taken on fresh fracture surface.

Table 2.12. Summary of Auger Results for Sample 760184 (MHT-92, with Weld)

Chart	Spot	x/t	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 54 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{C}, 272 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$	$\frac{I(\text{O}, 510 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
A761227 C-08 ^a	1	0.63	0.12	0	0.11	0
-09 ^a	2	0.04	0.10	0.64	0.44	0.22
A761228 C-01 ^a	3	1.00	0.06	0	0.35	0.20
-02 ^a	4	0.04	0.07	0.37	0.39	0.22
-03 ^a	5	0.04	0.01	0.52	0.41	0
-04 ^a	6	0.07	0.04	0.62	0.37	0.28
-05 ^b	5	0.04	0	0	0.62	0
-06 ^b	6	0.07	0	0	0.50	0

^aSpectra taken on fresh fracture surface.

^bSputtered for 2 min. Sputtering conditions were set to remove the equivalent of one atom layer of Ta₂O₅ per minute of sputtering time.

Fig. 2.10. A Sample Current Image of the Fracture Surface of Sample 760185, Showing the Spots Where Auger Spectra Were Taken.

Fig. 2.11. A Sample Current Image of the Fracture Surface of Sample 760184, Showing the Spots Where Auger Spectra Were Taken.

Table 2.13. The various spots that were analyzed were grouped according to their values of x/t . Spots with $x/t \leq 0.25$ were classified as being in the inner edge and those with $x/t \geq 0.75$ were grouped in the outer edge. All other spots were grouped in the middle region. Table 2.13 shows the average peak height ratios for MHT-90, -91, and -92 according to region, with the available data from MHT-103 and -104 for comparison. As with any condensation of data, some information is lost, and reference should be made to Tables 2.7 through 2.12 for details.

Table 2.13. Summary of Auger Results on the Distribution of Phosphorus and Thorium

Alloy	Region	$\frac{I(\text{Th}, 67 \text{ eV})}{I(\text{Ir}, 54 \text{ eV})}$	$\frac{I(\text{P}, 120 \text{ eV})}{I(\text{Ir}, 229 \text{ eV})}$
MHT-90, -91, and -92	Outer	0.052	0.029
	Middle	0.079	0.012
	Inner	0.056	0.708
MHT-103 and -104	Outer	0.050	0
	Middle	0.043	0.869
	Inner	0.020	2.234

Table 2.13 clearly indicates the high P concentration in the inner edge relative to the middle region and top edge. It also shows that the average P concentration in the inner edges of MHT-103 and -104 was more than three times as large as the average for the inner edges of MHT-90, -91, and -92. The difference between the average peak height ratios for the middle regions and top edges of MHT-90, -91, and -92 may not be statistically significant since the standard deviation from the average was larger than the averages themselves.

The averages of the thorium peak height ratios in Table 2.13 show a slight indication that the middle region of the fracture surface may be richer in thorium than the bottom and top edges. Small and large thorium peaks were found at both edges and the centers of the fracture surfaces. Analysis of the thorium peak was complicated by oxygen contamination, the small size of the thorium peak, and the strongly sloping background. Additional scatter may result from thorium having different binding

energies for the different grain boundaries making up the fracture surface. Because of the large amount of scatter in the results for thorium, the differences between the outer edges, middle regions, and inner edges of MHT-90, -91, and -92 may not be statistically significant.

The sulfur observed on MHT-91 and -92 is puzzling. Many metals are strongly embrittled by sulfur, yet MHT-91 and -92, which had significant contamination, were superior to MHT-103 and -104 which showed no sign of sulfur contamination. Additional analysis will be necessary if the significance of the sulfur on these samples is to be understood.

The one factor common to all of the samples described in Table 2.5 is the contamination of the grain boundaries near the inner edge of the fracture surface with P. The ratios of $I(P, 120 \text{ eV})$ to $I(\text{Ir}, 229 \text{ eV})$ are plotted as a function of x/t in Fig. 2.12 for all of the samples in Table 2.5. Values from MHT-103 and -104 are plotted as solid symbols and values from MHT-90, -91, and -92 are plotted as open symbols. Samples from MHT-103 and -104 clearly have more extensive P contamination than samples from MHT-90, -91, and -92. This makes it seem likely that the increased P contamination in MHT-103 and -104 is responsible for the cladding failure.

2.4.2 Analysis of Precipitate-Matrix Interfaces in Ir-0.3% W-0.1% Th Alloys

Auger electron spectroscopic analysis of the interface between thorium-rich precipitates (possibly Ir_5Th) and the Ir-W-Th solid solution has been obtained using an improved scanning Auger microprobe.* This instrument can obtain AES analysis using primary electron beams as small as $0.5 \mu\text{m}$ in diameter, hence making it possible to analyze the surface of precipitate particles on the intergranular fracture surfaces of the Ir-0.3% W-0.1 Th alloy. The sample used for this purpose (760160) had been heat treated 1 hr at 1500°C .

This analysis was undertaken in order to determine whether the chemistry of the precipitate-matrix interface is significantly different from the chemistry of the grain boundary. This information is required in order to evaluate the results of previous AES analysis using primary electron beams that are too large to allow individual precipitates to be resolved.

*Model 590 Scanning Auger Microprobe, manufactured by Physical Electronics Industries.

Fig. 2.12. A Plot of the Ratio $I(P, 120 \text{ eV})/I(Ir, 20 \text{ eV})$ as a Function of the Ratio x/t for All Samples on Table 2.5. Open symbols represent data from MHT-90, -91, and -92 that had acceptable impact performance; solid symbols represent data from MHT-103 and -104 that failed the simulant fuel impact tests.

Auger analysis using these larger electron beams results in an average over regions partially covered with precipitate particles.

Figure 2.13 shows two partial Auger spectra, one from a precipitate surface, and the other from a spot on the adjacent grain boundary fracture surface. A secondary electron image, including this portion of the fracture surface, is shown in Fig. 2.14. The ratio of I(Th, 67 eV) to I(Ir, 54 eV) is about 0.27 for the spectrum taken on the precipitate as compared to a ratio of about 0.09 for the spectrum taken on the region adjacent to the precipitate. Several other precipitates were similarly examined, and while there was considerable variability in the peak height ratios, the average for analysis taken on the precipitates is about 0.26 while the average for analysis taken on the grain boundary fracture surfaces is about 0.09. Complete Auger spectra taken on one of the precipitates did not indicate any other significant differences between the precipitates and the grain boundaries.

These results suggest that while the precipitate surfaces are richer in thorium than the grain boundary surfaces, the difference is probably not so large as to require correction of analysis where individual precipitates were not spatially resolved.

2.5 References

1. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. March 31, 1976*, ORNL/TM-5482.
2. Correspondence from E. A. Walker, MRC to A. C. Schaffhauser, ORNL, "Weekly Highlights, Heat Source Branch, NRA, Week Ending November 19, 1976."
3. E. M. Cramer and S. S. Hecker, *Postimpact Examinations of Three DOP 4 Iridium Shells from Simulant Fuel Sphere Assemblies*, LA-6176-MS, February 1976.
4. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. Sept. 30, 1976*, ORNL/TM-5810.
5. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. March 31, 1976*, ORNL/TM-5482.
6. L. E. Davis et al., *Handbook of Auger Electron Spectroscopy*, 2nd, Physical Electronics Industries, Inc., Eden Prairie, Minnesota.

ORNL-DWG 77-3534

Ir + 0.3 %W + 0.1 % Th

HEAT WTh-2

1hr/1500 °C

FRESH FRACTURE SURFACE

Fig. 2.13. Partial Auger Spectra from a Sample (760160) of Ir-0.3% W-0.1% Th Alloy. Spectrum A was taken on a precipitate and spectrum B was taken on the grain boundary fracture surface near the precipitate.

Fig. 2.12. A Plot of the Ratio $I(P, 120 \text{ eV})/I(\text{Ir}, 20 \text{ eV})$ as a Function of the Ratio x/t for All Samples on Table 2.5. Open symbols represent data from MHT-90, -91, and -92 that had acceptable impact performance, and solid symbols represent data from MHT-103 and -104 that failed the simulant fuel impact tests.

3. CURIUM-244 FUEL DEVELOPMENT*

3.1 Introduction

Curium-244, which is present in irradiated nuclear fuel and is the waste from fuel recovery, has potential for use as a fuel in radioisotope heat sources. The objectives of this program are (1) to investigate potential methods for recovery of Cm-244 from power reactor fuel wastes and (2) to determine the compatibility of $^{244}\text{Cm}_2\text{O}_3$ with materials which might be suitable as fuel encapsulants in such heat sources. To accomplish these objectives, we are initiating compatibility tests of $^{244}\text{Cm}_2\text{O}_3$ with various high-temperature alloys.

3.2 Compatibility Test Matrix

J. R. Keiser and J. R. DiStefano

Preparations have resumed for the $^{244}\text{Cm}_2\text{O}_3$ compatibility testing program which was terminated in FY 1974. The test program calls for tensile specimens of selected alloys to be aged in contact with $^{244}\text{Cm}_2\text{O}_3$. Alloys to be tested and their exposure conditions are given in Table 3.1. Most specimens and their capsule components were fabricated before the program was terminated, and these are still available. Three new alloys (noted in Table 3.1) will replace some of the previously selected alloys.¹ Specimens of these new alloys will be fabricated when the technique for fabricating the required rectangular Cm_2O_3 pellets is demonstrated.

3.3 Fuel Specimen Fabrication

C. L. Ottinger

After an evaluation of the operational status of the Curium Source Fabrication Facility (CSFF), it was decided to investigate alternative facilities and methods for preparing the rectangular fuel pieces needed for the

*Progress on work performed under Activity No. KJ 06 02 02 0, 189a No. 01367.

Table 3.1. Revised Test Matrix for Cm_2O_3 Compatibility Tests

500 hr/900°C Argon-Graphite	5000 hr/1100°C Vacuum-Graphite	10,000 hr/1400°C Vacuum-Graphite
Pt-3008/TZM ^a	Pt-3008	Ir-0.3% W ^b
Mo-46% Re	Mo-46% Re	Mo-46% Re
Ta-10% W	Ta-10% W	Ta-10% W
Hastelloy S ^b		
Inconel 617 or Alloy 556 ^b		

^aOuter container.^bNewly selected alloy.

compatibility/tensile strength tests. The Isotope Research Materials Laboratory (IRML) of the Solid State Division, ORNL, has been very successful recently in preparing ceramic materials by an extrusion technique, and fabrication of the rectangular $^{244}\text{Cm}_2\text{O}_3$ pellets can probably be done by this method. The ^{244}Cm experimental glove box line is not currently being used and has the essential services. Since refurbishing of the CSFF would be very expensive, and since experience with direct hot pressing of $^{244}\text{Cm}_2\text{O}_3$ in rectangular form has not been good, a decision was made to attempt the fabrication by extrusion in the glove box facility.

Test dies are being fabricated and the needed equipment is being checked and repaired as necessary. The first experimental pressings should be made early in February to determine if the technique can be used. If the tests are successful, the production dies will need to be fabricated to yield the exact size of pellets required. It will be necessary to fabricate pellets one at a time due to radiation limitations, but this is considered preferable to the high-expense approach of the CSFF.

3.4 References

1. E. Lamb, *ORNL Isotopic Power Fuels Quart. Progr. Rep. Dec. 31, 1973*, ORNL-4939, p. 5.

4. SELENIDE ISOTOPE GENERATOR SYSTEM SUPPORT*

4.1 Introduction

This program provides materials support for the Selenide Isotope Generator (SIG) System to the systems contractors (Teledyne Energy Systems and 3M Company). Currently, emphasis is on providing the materials, components, and technology required for the SIG Ground Demonstration System (GDS), scheduled for operation in 1978. The three major task areas are (1) Isolation Hot Frame (IHF) fabrication development, (2) compatibility and property evaluation of the Pt-3008 alloy selected for the IHF with the system materials and environments, and (3) characterization of proposed thermal insulation materials. The IHF is designed to isolate the isotope heat source environment from the selenide thermoelectric module portion of the system. The Pt-3008 alloy (Pt-30% Rh-8% W) was selected for the IHF because of its compatibility with graphite and good creep strength. We are providing material and technology for the development and component fabrication of the IHF cylinder and bellows. The task on compatibility includes evaluation of the effects on the mechanical properties of Pt-3008 of potential interactions with other system components such as insulations and selenide thermoelectric materials. The insulation evaluation task includes characterization of the microstructural and chemical composition, thermal stability, and compatibility with graphite.

4.2 Isolation Hot Frame Fabrication Development

R. L. Heestand

We are assisting Teledyne Energy Systems by developing fabrication techniques and providing the Pt-3008 hardware associated with the Isolation Hot Frame (IHF) for the Selenide Isotope Generator System. This consists of a cylindrical can and bottom approximately 188.8 mm (7.43 in.) OD $\times$ 139.7 mm (5.50 in.) high $\times$ 0.38 mm (0.015 in.) thick, a bellows 0.28 mm

*Progress on work performed under Activity No. KJ 06 01 01 0, 189a No. 01405 and Activity No. KJ 07 02 02 0, 189a No. 01320.

(0.11 in.) thick attached to the top of the cylinder, and transition joints for attachment to the exterior shell. The size of the components require sheet of a greater size than had been rolled previously. Sheet rolling development is described in another section of this report.¹

The bellows design requires relatively sharp and close tolerances. For this reason, the sheet material for the bellows was supplied to a commercial fabricator, Metal Bellows Corporation, who have the necessary equipment for the operation. Sheet from both arc-cast and extruded Pt-3008 and induction-melted Pt-3008 was supplied. It was intended that the induction-melted material, which had not been qualified for hardware, be used for practice forming, and the arc-cast and extruded materials be used for prototype hardware.

At this time, tooling has been completed for forming the bellows and practice bellows from stainless steel have been formed to check the tooling. Since low-ductility is anticipated in the seam weld of the Pt-3008,² it is planned to introduce work by planishing the weld and stress relieve to provide a better structure for forming. In addition, since increasing the temperature to 200°C increases the ductility of Pt-3008 sheet, it is planned to form the bellows at this temperature. Due to the limited amount of material and the additional time required for bellows fabrication, fabrication development of the cylinder and bottoms of the hot frame will be initiated upon receipt of sheet material from the third scale-up ingot.

4.3 Compatibility Studies

J. R. Keiser

4.3.1 Initial Compatibility Screening Tests

Utilization of Pt-3008 for the Isolation Hot Frame in the Selenide Isotope Generator System requires that compatibility of Pt-3008 with the various system components be demonstrated. From an assessment of the potential reactions of the many materials expected to be used in this system, we concluded that there were three which should be quickly evaluated for their effect on Pt-3008: the thermoelectric material Cu_2Se which has a rather high selenium partial pressure at elevated temperatures; the insulation Fiberfrax HiFi 660 which is composed of Al_2O_3 and SiO_2 , but

which has rather high concentrations of impurities including B, Na, and Fe which could detrimentally affect the Pt-3008, and the Min-K TE 1400 insulation which, in combination with graphite, could undergo the reaction

under the proposed operating conditions. Previous research has shown that silicon can diffuse into Pt-3008 along the grain boundaries and can cause complete loss of ductility at 1316°C.

The test program with exposure conditions is given in Table 4.1. Following exposure, the strength and ductility of the specimens were measured and compared with that of control specimens aged for the same time and temperature. The specimens exposed to the Min-K insulation/graphite combination showed no degradation of properties, but specimens exposed to Cu₂Se and specimens exposed to Fiberfrax insulation showed significant losses in strength and ductility. Typical results for the specimens exposed to Fiberfrax showed losses in tensile strength of at least 20% and as much as 80%, and losses in ductility of at least 50% and as much as 95% when compared with the control specimens. Metallographic examinations show a shift to an intergranular fracture mode. Examination of a fracture surface by means of Auger electron spectroscopy revealed approximately 5 at. % of both S and P. Sputtering results indicated that these elements existed only in a thin layer of the fracture surface. The specimens exposed to the Cu₂Se underwent a more severe degradation than did Fiberfrax-exposed specimens. Tensile strength losses ranged from 30 to 95% and the ductility was 50 to 100% less than it was for the control specimens. Metallographic examination indicated an intergranular fracture mode, and Auger examination showed a thin layer of selenium on the fracture surface. A more complete discussion of the Auger results is given in the next section.

These initial compatibility tests were intended to quickly screen some of the potential interactions under the "worst case" conditions. For this reason, materials on hand were used, and we made no attempt to optimize the pretest outgassing of the materials. As a result of this less than sufficient outgassing, the compatibility test results may indicate a more severe degradation than would be expected to occur with well outgassed starting material.

A follow-up series of compatibility tests is planned which will use the second scale-up heat of Pt-3008 whose properties are better defined and

Table 4.1. Selenide Generator Compatibility Tests Conducted

Component	Conditions	Time (hr)	Temperature (°C)
Fiberfrax HiFi 660 insulation	In contact with Pt-3008	1000	800, 950, 1100 ^a
Min-K in contact with graphite	In vicinity of Pt-3008	1000	800, 950, 1100
Cu ₂ Se	In vicinity of Pt-3008	1000	800, 950, 1100 ^b

^aThe 1100°C capsule burst after short exposure.

^bThe 1100°C capsule broke after 850 hr.

are typical of the material that will be utilized in the GDS. Materials to be tested with the Pt-3008 are (1) well outgassed Fiberfrax, (2) fibrous Al₂O₃ or the current choice for high-temperature insulation, and (3) Cu₂Se with its temperature carefully controlled. In addition, the effectiveness of graphite barriers in preventing interactions will be evaluated. It is planned that some of these tests will be started by February 1977.

4.3.2 Microanalysis Characterization — C. L. White

4.3.2.1 Exposure of Pt-3008 to Vapor from Cu₂Se. — A fractured tensile sample of Pt-3008 that had shown low ductility in a tensile test was received from J. R. Keiser. This sample had been heat treated 1000 hr at 950°C in an evacuated quartz tube that also contained a sample of Cu₂Se to simulate potential interactions with the selenide thermoelectric elements.*

The shorter of the two pieces (sample 760181) from the tensile sample was submitted for scanning electron microscopy (SEM), and a piece from the gage section of the larger piece was used to prepare a sample (760190) suitable for Auger analysis. Figure 4.1 shows the scanning electron micrographs of sample 760181. This sample clearly failed in an intergranular fashion, and showed some interesting faceting on the grain boundaries at points where three or four grains join together. From the appearance of the scanning micrographs, this faceting may be the result of some kind of corrosive

*The Pt-3008 and Cu₂Se were encapsulated in such a way as to prohibit physical contact between them and still allow a common environment.

Fig. 4.1. Scanning Electron Micrographs of the Fracture Surface of Sample 760181. This Pt-3008 tensile sample had fractured in a brittle fashion after exposure to Cu_2Se vapor for 1000 hr at 950°C . Note the faceted appearance of the fracture surface at triple points.

attack at the triple points, resulting in the loss of Pt, Rh, and/or W and the creation of an open network along the triple points. Optical metallography of a sample of Pt-3008 that has had a similar exposure to Cu_2Se is shown in Fig. 4.2. These photomicrographs also indicate the presence of an open network along the triple points.

The Auger spectrum from the fresh fracture surface of sample 760190 is shown in Fig. 4.3. The ratio $I(\text{Se}, 1318 \text{ eV})/I(\text{Pt}, 1970 \text{ eV})$ is 0.48. This is indicative of a $X_{\text{Se}}/X_{\text{Pt}}$ ratio of about 0.09.* After sputtering the fracture surface for 2 min with inert gas ions,[†] the ratio $I(\text{Se}, 1318 \text{ eV})/I(\text{Pt}, 1970 \text{ eV})$ was reduced to 0.11. The spectrum in Fig. 4.3 also shows small peaks due to 92 eV Si and 510 eV O.

These results suggest that the extreme brittleness of Pt-3008 after exposure to Cu_2Se at high temperatures may result from contamination of the grain boundaries by Se and/or the formation of an open network along the triple points which would have the effect of reducing the load-bearing cross section of the tensile sample. Additional analysis of more carefully prepared samples is planned in order to gain additional insight into this problem.

4.3.2.2 Exposure of Pt-3008 to Fiberfrax Insulation. — A fractured tensile specimen of Pt-3008 that had been heated 1000 hr at 950°C in contact with Fiberfrax insulation in a sealed quartz tube was received from J. R. Keiser. This specimen had failed in a brittle fashion during a tensile test at 950°C , exhibiting an ultimate tensile strength of 22,019 MPa and elongation of 2.9%. Scanning electron micrographs of the fracture surface on the smaller of the two pieces of this specimen (sample 760195) are shown in Fig. 4.4.

A small piece from the gage section of this tensile sample was used as an Auger sample (760194) which was analyzed in much the same way as sample 760190 (which was described previously). Figure 4.5 shows the Auger

*See note on p. 39 of this report concerning the conversion of peak height ratios to atom fraction ratios.

[†]Sputtering conditions were set so that the removal rate on Ta_2O_5 would be about one atom layer per minute.

Fig. 4.2. Optical Photomicrograph of a Sample of Pt-3008 Having Same Exposure to Cu_2Se as Sample 760181.

Fig. 4.3. An Auger Spectrum from the Fresh Fracture Surface of Sample 760190. This sample comes from the same tensile specimen as sample 760181.

Fig. 4.4. Scanning Electron Micrographs of Sample 760195. This sample of Pt-3008 alloy was heated 1000 hr at 950°C in contact with Fiberfrax insulation in an evacuated quartz capsule.

Fig. 4.5. An Auger Spectrum from the Fresh Fracture Surface of Sample 760194, Which Was Identical to Sample 760195 in Fig. 4.4.

spectrum taken from the freshly fractured sample. Significant concentrations of P and S are indicated by peaks at 120 and 150 eV, respectively, in Fig. 4.5. The $I(P, 120 \text{ eV})/I(Rh, 302 \text{ eV})$ ratio in Fig. 4.5 is 0.12, which suggests a X_P/X_{Rh} ratio of about 0.20.* The 152-eV S and 150-eV Pt peaks are overlapping in Fig. 4.5; the $I(S, 152 \text{ eV})/I(Rh, 302 \text{ eV})$ ratio is estimated to be about 0.15, which suggests a X_S/X_{Rh} ratio of about 0.15. Inert ion sputtering of the fracture surface for 2 min, and subsequent reanalysis indicated that the S- and P-rich region is within a few atom layers of the grain boundary.

These results suggest that the brittleness of Pt-3008 after exposure to Fiberfrax insulation at 950°C may be due to contamination with P and/or S. Examination of a reference sample (having the same thermal history, but without contact with Fiberfrax) is planned in order to determine the source of the P and S.

4.4 Insulation Characterization

G. C. Wei

The assessment of commercially available thermal insulation materials for the selenide generator was completed and is being reviewed for publication. Fibrous insulation materials which have the potential to be used as load-bearing or non-load-bearing thermal insulations were reviewed, and thermophysical properties supplied by manufacturers or published in the literature were presented. Major conclusions are summarized below:

1. Manufacturers' thermal conductivity data should only be used as a guideline.
2. Thermal conductivity must be measured in an environment simulating application conditions.
3. Compatibility between fibrous insulations and graphite, Pt-3008, and thermoelectric elements must be confirmed before the GDS tests.
4. Further work on fibrous insulation structural and microstructural changes at high temperatures for long-term operation is needed.

*See the discussion on p. 39 of this report concerning calculations of atom fraction ratios.

5. Procedures of outgassing, forming, and installation of fibrous insulations are not fully established.

Several fibrous insulation materials were characterized with the aid of SEM and x ray. The results of SEM and x-ray characterization on Cotronics 360 are summarized as follows:

1. An extraneous crystalline phase containing Mg, Ca, and Si is present and amounts to about 1 vol %. It is expected that this will cause shrinkage and thus structural instability, which in turn, will tend to increase the thermal conductivity.

2. Impurities such as Ca, Mg, Fe, Ti, Cu, In, Se, and Mn are present in appreciable amounts.

3. A large quantity of shot content is present and the shot is not distributed uniformly in the insulation.

The above findings suggest that Cotronics 360 is not a desirable fibrous insulating material for the heat source of the selenide generator.

Min-K TE-1400 consists of silica particles of 15- to 20- μ m size. Minor concentration of TiO_2 opacifier was detected. Silica glass fibers are used as structural reinforcement in Min-K TE-1400. Fiberfrax HiFi 660H paper is made up of amorphous alumina-silica fibers. Unfiberized shot is present in Fiberfrax HiFi 660H. Kaowool 3000 is based on alumina fibers in which a slight amount of silica is also present. Binder phase consists of alumino-silicates. Unfiberized shot content is zero and the distribution of fiber diameter, length, and orientation is rather uniform in Kaowool 3000. Zircar ZYFB3 consists of hafnium-stabilized zirconia fibers. Significant grain growth has occurred in each Zircar fiber, and fibers are present in the form of bundles. These observations indicate that these insulations may not be stable during long-term operation at high temperatures.

4.5 References

1. See section 1.3.1, "Pt-3008 Scale-Up and Fabrication Development," p. 13 of this report.
2. A. C. Schaffhauser, *Isotopic Power Materials Development Quart. Progr. Rep. June 30, 1976*, "Welding of Pt-3008," ORNL/TM-5569, p. 12.

5. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT*

5.1 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.

5.2 C-103 Gaseous Interactions

5.2.1 Creep Properties of C-103 in Contaminating Environments - H. Inouye

A C-103 sheet specimen has been creep tested under a sequence of controlled environmental and stress conditions to determine the effect of pyromellitic acid on its creep rate at 878°C. Pyromellitic acid ($C_{10}H_6O_8$) is a principal compound outgassed from the alternator-stator at its operating temperature of 121°C. As reported previously,¹ this compound decomposes at the test temperature to H_2O as the major gas species with smaller fractions of CO , CO_2 , and O_2 .

*Progress on work performed under Activity No. KJ 07 02 01 0, 189a No. 01319.

The creep rates measured for C-103 for several conditions are tabulated in Table 5.1. These data show that the creep rate of an uncontaminated specimen (condition A) is less than 10% of the rate measured in the $C_{10}H_6O_8$ environment at 1.3×10^{-3} Pa (10^{-5} torr) (condition B). Although it is believed that the strain measured for the test interval under the latter condition is caused mainly by dilation of the specimen during its contamination, we were unable to confirm this when the load was removed from specimen. Under no-load conditions (condition C), the creep rate for the 1230–1906 time interval was only $1.3 \times 10^{-4}\%/hr$ and not the expected higher rate of approximately $4 \times 10^{-3}\%/hr$; however, it was noted that on reapplying the load in a vacuum (condition D), the creep rate was near the expected dilation rate but only for 94 hr.

Table 5.1. Effect of $C_{10}H_6O_8$ on the Creep Rate of C-103
Stressed to 145 MPa (21 ksi) at $878^\circ C^a$

Test Condition	Environment	Test Interval ^b (hr)	Creep Rate ($10^{-4}\%/hr$)	Remarks
A	1.3×10^{-6} Pa (10^{-8} torr) vacuum	0–753 (753)	3.5	stressed
B	1.3×10^{-3} Pa (10^{-5} torr) $C_{10}H_6O_8$ ^c	753–1230 (477)	39.0	stressed
C	1.3×10^{-3} Pa (10^{-5} torr) $C_{10}H_6O_8$	1230–1906 (676)	1.3	no load
D	1.3×10^{-6} Pa (10^{-8} torr) vacuum	1906–2000 (94)	42	stressed
D	1.3×10^{-6} Pa (10^{-8} torr) vacuum	2000–2675 (675)	2.2	stressed
D	1.3×10^{-6} Pa (10^{-8} torr) vacuum	1906–2675 (769)	7.1	stressed

^aGage dimensions: $50.80 \times 3.2 \times 0.762$ mm ($2.0 \times 0.125 \times 0.030$ in.). Vacuum annealed 3 hr at $1650^\circ C$.

^bParentheses () indicate time at test condition.

^cPyromellitic acid.

The strain-time curve for the specimen tested under the conditions outlined in Table 5.1 is shown in Fig. 5.1. Distinct breaks in the curve occur precisely at the time of each condition change. Since the sharp break in the curve observed when the load is removed at 1230 hr is not consistent with prior dilation data, we assume that it was caused by some bending of the specimen when the load was removed, then, on reapplying the load at 1906 hr, the specimen straightened out over the next 94 hr. With this interpretation, the creep rate of the specimen of $2.2 \times 10^{-4}\%/hr$ between

ORNL-DWG 77-2763

Fig. 5.1. Creep Curve for C-103 at 878°C Under Various Environmental and Stress Conditions.

2000–2675 hr represents the creep properties of a predoped C-103 specimen; and as the data in Table 5.1 indicates, is significantly stronger than an uncontaminated specimen where rate is $3.5 \times 10^{-4}\%/hr$. Another interpretation of these data is that the synergistic effects of stress and contamination is greater than the sum of each of these factors alone. To resolve which of these interpretations is representative of the data, the creep test will be repeated but at a low stress.

5.2.2 Kinetics of Gaseous Absorption in C-103 — C. T. Liu and H. Inouye

To study the kinetics of gaseous absorption in C-103, sheet specimens with a thickness of 0.80 mm (0.0315 in.) were vacuum annealed 1 hr at 1650°C then exposed to carbon monoxide gas at a pressure of 1.3×10^{-3} Pa (10^{-5} torr) at 1000 and 850°C . The specimens were periodically removed from the furnace and weighed.

The specimens showed no clear indication of surface film after exposure. The exposed specimens exhibited weight gains due to absorption of oxygen and carbon from the environment. Figure 5.2 shows the plot of the weight gain as a function of square root of exposure time (t). The weight gain measured from tensile specimens [with a thickness of 0.64 mm (0.025 in.)] exposed at 1.3×10^{-3} Pa (10^{-5} torr) CO at 850°C is also plotted as curve C in Fig. 5.2. The difference between curves B and C is due to thickness effect. As shown in the plot, the weight gain in C-103, after an incubation period, increases linearly with $t^{1/2}$. This clearly indicates that a diffusion process controls the kinetics of carbon monoxide gas absorption in C-103 at 850 and 1000°C . Because of the diffusion control, the absorption rate decreases continuously with exposure time. After a 500-hr exposure to 1.3×10^{-3} Pa (10^{-5} torr) CO, the C-103 sheet specimens were contaminated at a rate of approximately 4.4 ppm/hr at 1000°C and approximately 1.8 ppm/hr at 850°C .

5.2.3 Tensile Properties of C-103 in Contaminating Environments — C. T. Liu and H. Inouye

To study the embrittlement effects of gaseous environments, 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 (10^{-5} torr)

Fig. 5.2. Plot of Weight Gain in C-103 Specimens Exposed to 1.3×10^{-3} Pa (10^{-5} Torr) CO as a Function of Square Root of Exposure Time (t). Curve A: exposed at 1000°C, specimen thickness: 0.8 mm (0.0315 in.); curve B: exposed at 800°C, specimen thickness: 0.8 mm (0.0315 in.); curve C: exposed at 850°C, specimen thickness: 0.64 mm (0.025 in.).

at 1000 and 850°C. To date, specimens contaminated to a level of 4200 ppm have been tested at room temperature, 800 and 1000°C. The tensile elongation is plotted as a function of weight gain in Fig. 5.3. The specimens exposed to carbon monoxide at 1000°C showed a steady decrease in ductility with weight gain at all three test temperatures. In contrast, the ductility of the 850°C-exposed specimens decreases sharply to a level of 1000 ppm, then increases slightly in the range 1000–3400 ppm, and finally decreases again beyond 3400 ppm. The C-103 specimens contaminated to 4200 ppm at 850°C still had 3–5% elongation at room temperature and 850°C.

Numerous cracks were observed in the surface of the tensile-fractured specimens contaminated with more than 1000 ppm. Examination of the fracture surface revealed that it generally consisted of two fractured regions, i.e., a ductile region in the center surrounded by brittle fracture at the rim. This observation also indicates an interstitial gradient with a higher level of contamination near the specimen surfaces.

5.3 Welding and Brazing

G. M. Goodwin and J. D. Hudson

We are testing lap-shear specimens of Hastelloy X brazed to itself with Palniro 7 (Au–8% Pd–22% Ni) and Hastelloy X brazed to C-103 alloy sheet using Coast Metals 50 (Ni–3.5% Si–2.5% B–1% Fe). Recent tensile tests are summarized in Table 5.2, with previously reported tests for comparison. Increasing strain rate by a factor of 10 (from 0.05 to 0.5/min) appears to show somewhat higher strength and ductility in the Hastelloy-to-Hastelloy brazes at 649°C.

A repeat in argon of a test previously run in air showed improved strength and ductility for a Hastelloy-to-columbium alloy braze at 732°C. Subsequent tests involving the C-103 alloy at elevated temperatures will be performed in argon.

Braze specimens of both types have been aged 1000 hr at 732°C in vacuum, and tensile tests of aged specimens are being performed at 20 and 732°C.

Fig. 5.3. Plot of Tensile Elongation as a Function of Weight Gain for C-103 Specimens Exposed to Carbon Monoxide Gas at 1000 (Filled Symbols) and 850°C (Open Symbols).

Table 5.2. Tensile Properties of Hastelloy X and Hastelloy X to C-103 Alloy Brazements^a

Specimen Designation	Specimen Type ^b	Test Temperature (°C)	Ultimate Strength ^c MPa(ksi)	Total Elongation (%)	Fracture Location
H-3	H/H	649	148.1(21.5)	4.5	Braze
H-8	H/H-Fast	649	184.8(26.8)	6.7	Braze
HC-1	H/C	732	91.0(13.2)	4.0	Braze
HC-1-R ^b	H/C	732	129.3(18.8)	6.9	Braze

^aLap-shear specimens of 3.2-mm (0.125-in.) Hastelloy sheet brazed to itself with Palniro 7 alloy (Au-8% Pd-22% Ni) and to 2-mm (0.080-in.) C-103 alloy sheet with Coast Metals 50 alloy (Ni-3.5% Si-2.5% B-1% Fe), tested at strain rate of 0.05/min in air.

^bH/H = Hastelloy X to itself; H/C = Hastelloy X to C-103 alloy; Fast = 0.5/min strain rate; R = repeat in argon.

^cMaximum load dividend by overlap area.

^dBased on entire gage length of 5.1 cm (2 in.).

5.4 Reference

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6. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE*

6.1 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.

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

D. E. Harasyn

Production data on Ir-0.3% W forming disks shipped in October, November, and December of 1976 are shown in Table 6.1. In January 1977, two more shipments will be made to MRC to complete the forming disk requirement for the SIPS project.

Approximately 2060 cm² (320 in.²) of 0.25-mm- (0.010-in.-) thick and 2510 cm² (390 in.²) of 0.13-mm- (0.005-in.-) thick Ir-0.3% W foil has been shipped to Battelle Columbus Laboratories for vent experiments.

6.3 Grain Growth in Ir-0.3% W Alloys

D. E. Harasyn

In a recent monthly report from LASL,¹ very large grains were observed in fueled and impacted PICS made from DOP-4 Ir-0.3% W. MHFT-56 had 2-5 grains across the wall. MHFT-55 also had large grains near the inside of the shell, but appeared to have smaller grains near the exterior of the

*Progress on work performed under Activity No. KJ 07 03 00 0, 189a No. 01322.

Table 6.1. 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							
TR-512	4					1	1	1	1			10/6/76
-513	2					1	1					10/6/76
-514	4					1	1	1	1			10/6/76
-515	4			P		1	1		1			10/6/76
-516	4					1	1	1	1			10/6/76
-517	4					1	1	1	1	24 ^b	24	10/6/76
-518	4					1	1	1	1			10/26/76
-519	4				U	1	1	1				10/26/76
-520	4				U	1	1	1				10/26/76
-521	4					1	1	1	1			10/26/76
-522	4			U		1	1		1			10/26/76
-523	4					1	1	1	1	22 ^c	46	10/26/76
-524	4	P					1	1	1			11/12/76
-525	4					1	1	1	1			11/12/76
-526	4	P		P			1		1			11/12/76
-527	4		U			1		1	1			11/12/76
-528	4			P	U	1	1					11/12/76
-529	4	U		D	D		1	1	1			11/12/76
Reject Lot ^d										17 38	63 101	12/6/76
TR-530	4	U					1	1	1			12/20/76
-531	4					1	1	1	1			12/20/76
-532	4					1	1	1	1			12/20/76
-533	4					1	1	1	1			12/20/76
-534	4					1	1	1	1			12/20/76
-535	4		U			1		1	1	25 ^e	126	12/20/76

^aP = dye penetrant; U = ultrasonic; and D = dimensional.

^bShipment included reground disks TR-507 #1, TR-508 #4, and TR-511 #2.

^cShipment included reground disk TR-515 #3.

^dLot consisted of previously retained disks, some of flight hardware quality, many with minor defects.

^eShipment included reground disks TR-526 #1 and #3, and TR-528 #3.

shell. MHFT-55 had 3-12 grains across the wall. The heat treatment of these PICS in contact with PuO_2 fuel was 32 days at 1330°C plus 200 sec at 1550°C plus 1 hr at 1440°C . Since MHFT tests are made on fuel spheres taken from the MRC production line, MHFT-55 and -56 received processing heat treatments of 1 hr at 1175°C and 1 hr at 1500°C prior to heat treatment at LASL. At ORNL, we duplicated the LASL aging treatments in vacuum without the fuel using the same lot of iridium as in the MHFT tests (HD 403-408). The MRC heat treatments of 1 hr at 1175 and 1500°C were omitted, but their effect on grain growth was calculated from previous data. Doped (HD-405, DOP-4-4) and undoped (WG-202) Ir-0.3% W samples were taken from flat sheet stock and annealed in an iridium boat. The results shown in Table 6.2 are consistent with previous grain growth data obtained at ORNL.² Had our samples received the 1-hr heat treatment at 1500°C prior to the complex heat treatment, the calculated number of grains across the thickness would be 5.9 for WG and 12.9 for DOP-4. Very little data has been published on grain growth in Ir-0.3% W after it is hydroformed into hemishells. However, it is unlikely that the forming process significantly changes grain growth in the alloy since the forming is done while the material is in a fibrous cold-worked condition. Three data points are available from a report by Frank et al.³ on hydroformed WC (undoped) material (1500°C ; 1, 6, 18 hr). These three

Table 6.2. Comparison of Grain Sizes for
Ir-0.3% W Samples Exposed to
High Temperatures^a

Alloy Designation	Ingot Number	Number of Grains Across a 0.64-mm (0.025-in.) Thickness	
		Observed	Calculated ^b
Ir-0.3% W	WG-202	6.8	6.4
DOP-4	HD-405	13.2	—
DOP-4	DOP-4-4	13.4	14.7

^aHeated 768 hr at 1330°C plus 200 sec at 1550°C plus 1 hr at 1440°C in vacuum.

^bCalculated from data given in *Grain Growth in Ir-0.3% W Alloys*, ORNL-5233, by D. E. Harasyn and A. C. Schaffhauser.

points agree well with data obtained at ORNL on flat sheet materials. As yet, no satisfactory explanation has been given to explain the discrepancy between ORNL grain size data and MHFT-55 and -56.

Some representative microstructures from the DOP-4 Ir-0.3% W cladding of fuel simulant impact spheres MHT-103 and -104 are shown in Fig. 6.1. Nothing unusual was observed in the microstructures of the base metal. The spotty appearance of the weld zone contrasts with the clean microstructure of the base metal. The spots are thought to be etch pits.

Grain size data on MHT-103 and -104 are given in Table 6.3 along with data on DOP-4 production material which was annealed in vacuum at 1500°C for 19 hr. MHT-103 and -104 were annealed at General Electric Company 18 hr at 1500°C, loaded with ThO₂ fuel simulant, and then annealed one more hour at 1500°C. Although the grain size of the MHT samples are slightly smaller than those of the other DOP-4 samples, the difference is not considered significant.

6.4 Impact Properties of Production Material

C. T. Liu

We continue to qualify the impact properties of DOP-4-type Ir-0.3% W production material used for space nuclear flight systems hardware. Sheet specimens of heats HD-421, -446, HDR-469, and TR-500, heat treated 19 hr at 1500°C, then impacted at a standard condition of 85 m/sec (280 fps) and 1350°C. The impact results are presented in Table 6.4 together with those obtained previously from different lots of production material. The grain size of the impacted specimens was measured and is also reported in Table 6.4. The preparation of these materials is described in the footnotes of this table. The impact properties and grain size of heats HD-421, -446, and HDR-469 are not different from the other DOP-4 production lots. However, lot TR-500 had coarser grain structure and lower impact ductility as compared with the HD lots.

The impact elongation of production materials WGD, HD, and TR is plotted as a function of grain size in Fig. 6.2. The data are quite scattered in the plot; however, a general trend is still evident; that is, the impact ductility decreases with increasing grain size.

BASE METAL

WELD ZONE

MHT-104B

Fig. 6.1. Microstructures of MHT-103B and -104B. 100 $\times$.

Table 6.3. Grain Size of MHT-103B and
-104B as Compared to Various
DOP-4 Production Samples^a

Sample Number	Average Number of Grains Across a 0.064-cm (0.025-in.) Thickness
MHT-103B	11.7
-104B	11.9
HD-400	13.0
-403	9.1
-404	10.1
-406	10.3
-408	10.3
-412	10.9
-416	8.9
-424	9.0
-428	10.3
-436	10.4
-440	10.0
-459	9.1 ^b
HD average	10.1 ^b
WGD-266	7.8
-271	9.4
-274	8.4
-279	9.3
-290	10.0
WGD average	9.0 ^b

^aAll samples annealed 19 hr at 1500°C in vacuum except MHT-103B and -104B which are as-received from Mound Laboratory.

^bStandard deviation of the average is 0.2.

Table 6.4. 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
WC-65 ^c	3.4	9.6	21	Mainly GBS
WG-204 ^c	3.8	10.7	23	Mainly GBS
DOP-4-1 ^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-411 ^g	9.0	18.8	49	TF (Ma) and GBS (Mi)
HD-416 ^g	8.0	18.1	45	TF (Ma) and GBS (Mi)
HD-416 ^g	7.3	14.8	36	TF and GBS
HD-421 ^g	10.2	21.6	41	TF (Ma) and GBS (Mi)
HD-428 ^g	8.9	15.2	39	TF and GBS
HD-428 ^g	8.3	16.5	42	TF (Ma) and GBS (Mi)
HD-436 ^g	8.1	18.5	46	TF (Ma) and GBS (Mi)
HD-440 ^g	11.7	20.5	59	Mainly TF
HD-446 ^g	8.9	20.9	50	TF (Ma) and GBS (Mi)
HD-459 ^g	10.7	18.9	57	Mainly TF
HDR-469 ^h	9.6	23.2	57	Mainly TF
TR-500 ⁱ	6.2	15.2	44	TF and GBS

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

^bGBS = grain-boundary separation; TR = 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.

^hIr-0.3% W alloy doped with DOP-4 dopants, produced from 100% HD recycled skulls and heads.

ⁱIr-0.3% W alloy doped with 30 ppm Th, produced from WA and WF scraps.

Fig. 6.2. Plot of Impact Elongation as a Function of Grain Size for Production Materials HD, TR, and WGD.

6.5 References

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2. D. E. Harasyn and A. C. Schaffhauser, *Grain Growth in Ir-0.3% W Alloys*, ORNL-5233 (January 1977), pp. 8-11.
3. R. G. Frank, A. C. Schaffhauser, W. Wyder, *Backup Iridium Hemisphere Program Task Force Report*, General Electric Space Division, GESP-744 (April 19, 1974), pp. 37-54.

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