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

A. C. Schaffhauser

MASTER

OAK RIDGE NATIONAL LABORATORY

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METALS AND CERAMICS DIVISION

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

A. C. Schaffhauser

JUNE 1976

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37830
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MASTER

FOREWORD

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

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

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

Isotopic Power Materials Development Quarterly Report
ORNL/TM-5285 Period Ending December 31, 1975

Isotopic Power Materials Annual Report
ORNL-TM-5116 Period Ending June 30, 1975

Isotopic Power Materials Development Monthly Reports

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

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

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SUMMARY

1. HIGH-TEMPERATURE ALLOYS FOR SPACE ISOTOPIC HEAT SOURCES

Continued studies of the beneficial effects of thorium additions to Ir-0.3% W alloys show that, in terms of the mechanical properties at low strain rate, the 200 ppm Th alloy (DOP-14) has optimum ductility and fracture behavior at 650°C (the fracture transition temperature). Results of grain growth studies at 1500 and 1800°C indicate that alloys containing 200-1000 ppm Th maintain a relatively fine grain microstructure after severe thermal exposure. Studies on the isolation of the important DOP-4 dopants show that aluminum, in addition to thorium, has a beneficial effect on the ductility, fracture transition, and grain growth behavior of iridium alloys. Long-term grain growth behavior of doped and undoped iridium alloys at 1300-1500°C for times to 2000 hr show that, for the longer times and higher temperatures, grain growth of these alloys approaches the ideal grain growth law with an exponent of $-1/2$ for the number of grain boundaries across the cladding thickness as a function of time. Two advanced iridium alloys containing 2% Ru with the DOP-4 dopants and 200 ppm Th additions have been fabricated for evaluation.

Fabrication and inspection of sheet from the first consumable vacuum-arc-melted scale-up ingot of the Pt-3008 alloy was completed, and 2.3 kg (5.1 lb) of cold rolled sheet was supplied to LASL for impact capsules and compatibility studies and 1.4 kg (3.1 lb) was distributed to ORNL experimenters for mechanical property and welding studies. Chemical analysis indicates slightly higher levels of rhodium in sheets rolled from the nose and tail of the extrusion as compared to the central section. Trace impurities are within acceptable limits. Microstructural and recrystallization studies were completed. Consumable vacuum-arc melting of a second scale-up ingot weighing 8.35 kg (18.4 lb) was completed. Prealloyed melting stock for a 5.6-kg (12.4-lb) commercial induction-melted ingot was also prepared. The room-temperature ductility of electron-beam and gas tungsten-arc welds in sheet from a small ingot of Pt-3008 annealed 1 hr at 1200°C is 2% vs 10% in the base metal. A postweld heat treatment of 1 hr at 1200°C increased the ductility of the electron-beam-welded specimens to 5%, but showed no significant effect on the ductility of the gas tungsten-arc-welded specimen.

Attempts to roll arc-melted and powder metallurgy ruthenium billets at 1300–1500°C were not successful due to formation of pinhole defects, possibly caused by the molybdenum cladding used for protection during rolling. Phase diagram studies confirm that the solubility of rhodium in ruthenium and ruthenium in rhodium is at least 20% as reported.

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

Tensile impact tests of developmental doped iridium alloys at 1350°C and 85 m/sec (280 fps) indicate that aluminum, in addition to thorium, is effective in improving the impact properties of Ir–0.3% W alloys. The alloys containing 40 ppm Al plus 30 ppm Th had about 27% elongation after annealing 19 hr at 1500°C vs 15% for an alloy having no aluminum and 10% for an undoped material. Higher thorium additions also increased the ductility to 24% for the 100 ppm Th alloy and 29% for alloys containing 200–1000 ppm Th for the same test conditions. After a severe heat treatment at 1800°C, the 200–1000 ppm Th alloys are clearly superior having 25% ductility vs 14% for an alloy containing 100 ppm Th, 11% for the DOP-4 alloy, and 2–5% for the undoped material. The 200 ppm Th alloy (DOP-14) also has better impact ductility at lower impact temperatures (1050°C): 29% vs 15% for DOP-4 and 5% for undoped material. The superior impact properties of the high-thorium alloys are due to second-phase particles which retard grain growth during high-temperature heat treatment. Long-term aging of tensile impact specimens of DOP-4 alloys and undoped iridium at 1330°C was continued. Impact tests at 1350°C and 85 m/sec (280 fps) on specimens aged 1000 hr confirm the continuing excellent impact ductility of the DOP-4 alloy as compared to undoped material (27% vs 8%). Extrapolation of the DOP-4 data indicates that this alloy may retain sufficient ductility (14%) for operating times of 10,000 to 100,000 hr.

Oxidation tests on Pt-3008 tensile specimens scheduled for 10,000 hr have completed about 4000 hr to date. After 3000 hr of exposure at 600°C, no weight change or effects on the tensile properties at 600°C were observed; however, at 800°C, there was a weight gain due to the formation of an oxide film, and the tensile ductility at 800°C was reduced from 27 to 1.6%. At 1000°C exposure, there was a weight loss and the ductility at 1000°C was 4.2%. Pt-3008 compatibility specimens exposed to ZrO₂ in vacuum

and helium for 1000 hr at 1050°C were tensile tested at 25 to 1316°C. Specimens exposed to ZrO₂ in vacuum had higher strength than control specimens at all test temperatures, possibly due to some reaction with ZrO₂; however, there was no indication of embrittlement. Specimens exposed to ZrO₂ in helium were significantly weaker than control specimens due to accelerated grain growth in the helium environment.

Additional Auger studies of grain boundary surfaces in Ir-0.3% W alloys with thorium additions to confirm that the segregation of thorium to concentrations of 3-5 at. % is independent of bulk thorium concentrations between 30-1000 ppm. Ion microprobe mass analysis of a cross section of the 1000 ppm Th alloy showed that the second-phase particles are iridium- and thorium-rich. Auger analysis of an additional iridium sample from a FSA impact sphere showed 5 at. % P on the grain boundary surface which was removed by inert ion sputtering less than 10 atom layers. Grain boundary analysis of several heats of Pt-3008 using Auger spectroscopy showed 0.5-1.5 at. % P and up to 2 at. % Si; however, there was no correlation between the impurity segregation and the ductility minimum at 1093°C.

Tensile tests at 24, 316, 760, and 980°C on Inconel 625 specimens aged 11,000 hr at 816°C were completed, complementing earlier tests conducted on Haynes 25, Haynes 188, and Hastelloy N specimens. The results were interpreted with the aid of metallographic and electron probe analyses of internal microstructures, x-ray diffraction identification of phases, and electron-beam scans of fracture surfaces. Long-term aging at 816°C did not alter the yield and tensile strengths significantly; however, large changes in the ductility were observed. Whereas aging substantially depreciated ductilities for tests conducted at room temperature, tests at 760°C (region of the ductility minimum for solution-annealed condition) unexpectedly gave a substantial improvement in ductility. This improvement in ductility was reflected in very high fracture toughness values at 760°C (2-3 times higher than at other temperatures) for the cobalt-base alloys. Aged Inconel 625 also gave high ductility and toughness at 760°C but, in addition, displayed high toughness at room temperature, being 2-3 times higher than for other alloys at this temperature. As a rule, high ductility is equated with intergranular fracture. The inversion of ductilities at 760°C, brought about by overaging at 816°C, is explained in terms of the secondary phases involved, how and when they precipitated, and their effects in depleting the matrix of tungsten.

3. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT

Work was initiated at the beginning of this quarter to determine the effects of potential gaseous impurities in the operating environment of the Brayton Isotope Power System (BIPS) on the mechanical properties of the C-103 alloy selected for the heat exchanger, hot ducting, and turbine plenum. Modification of an environmental creep apparatus to permit testing of C-103 sheet specimens was completed. In the interim, the existing equipment was used to test tubular specimens of T-111, which are chemically similar to C-103. Tests at 1000°C and 15.2 MPa (2.2 ksi) in an environment of 1.3×10^{-3} Pa (1×10^{-5} torr) air or carbon monoxide showed much higher strains than predicted from published vacuum creep data. Exposure of unstressed specimens to 1.3×10^{-3} Pa (1×10^{-5} torr) of oxygen at 1000°C resulted in a weight gain of 10,000 ppm impurities in 100 hr and produced a dilation of over 1%.

The status of high-temperature bimetallic joint development was reviewed, and preparation specimens for long-term aging were initiated. Specimens are also being prepared to determine the mechanical properties of a recuperator braze alloy.

4. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE

Fabrication of iridium forming disks was suspended early in the quarter due to the detection of microscopic surface delaminations in metallographic certification samples. The defects were typically 0.1-mm (0.004-in.) long and less than 0.01 mm (0.0004 in.) below the ground surface. Investigations indicate that the most likely sources of the defects are the grinding, specimen cutting, and/or metallographic preparation operations. The defects have not been completely eliminated; however, additional testing showed no detrimental effects on the formability or impact ductility. Therefore, the nonconformance was accepted and fabrication was resumed. Only 18 of a scheduled 60 disks were shipped during this quarter; however, material in process is sufficient to meet Mound Laboratory's requirements with no slippage in the heat source assembly schedule.

Development of rolling procedures to cold roll 0.12-mm- (0.005-in.-) thick iridium foil in greater widths was completed using the doped iridium alloy. Approximately 1400 cm² (210 in.²) was shipped to Mound Laboratory.

Tensile impact tests at 1350°C and 85 m/sec (280 fps) of a number of current production lots of forming disks show that the ductility after a 19 hr at 1500°C heat treatment is reduced from about 21% in the first production lot to $16.5 \pm 2\%$ in the fourth and sixth production lots. No detrimental effects of the microscopic surface delaminations were observed. The reduction in ductility is most likely due to the loss of aluminum dopants as the amount of recycled melting stock increases in the later production lots. However, the ductility of subsequent production lots should remain above 15% (the ductility of a doped alloy with no aluminum).

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

1.1 Introduction

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

1.2 Iridium Alloys

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

In order to study the effects of thorium content in the mechanical and metallurgical properties, Ir-0.3% W alloys have been doped with different levels of thorium. Two new thorium-doped alloys containing 500 and 100 ppm Th, designated as DOP-21 and -22, were prepared and fabricated into sheets using the current production schedule.¹ The tensile properties of DOP-21 and -22 and WTh-2 were determined at 650°C [at a crosshead speed of 2.54 mm/min (0.1 in./min)] after a heat treatment of 1 hr at 1500°C. The tensile results are presented in Table 1.1 and are compared with WTh-1, DOP-14, and undoped WG. The thorium content in these alloys is also listed in Table 1.1. The yield strength of the alloys increases continuously with thorium content. The tensile strength also increases with thorium content to 500 ppm, but decreases at a level of 1000 ppm (WTh-2). The DOP-21 alloy (doped with 500 ppm Th) had a 240% higher yield strength and a 40% higher

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

Table 1.1. Tensile Properties^a of Doped and Undoped Ir-0.3% W Sheet Specimens^b Tested at 650°C

Specimen	Nominal Dopant Concentration (ppm)	Strength, MPa(psi)		Elongation (%)	Fracture Mode ^c
		Yield	Tensile		
WG-202	None	51.1(7,400)	488.5(70,800)	30.1	Mainly GBS
WTh-1	50 Th	72.5(10,500)	536.8(77,700)	39.2	GBS (Ma) and TF (Mi)
DOP-22	100 Th	77.9(11,300)	551.2(80,000)	38.5	Mainly TF
DOP-14	200 Th	96.6(14,000)	609.1(88,400)	41.5	Mainly TF
DOP-21	500 Th	121.3(17,600)	672.5(97,600)	36.7	TF (Ma) and GBS (Mi)
WTh-2	1000 Th	126.1(18,300)	594.6(86,300)	27.0	TF (Mi) and GBS (Ma)
DOP-15	30 Th, 80 Fe	77.9(11,300)	507.1(73,600)	40.1	Mainly TF
DOP-13	30 Th, 40 Al	86.8(12,600)	557.4(80,900)	40.2	Mainly TF
DOP-16	30 Th, 40 Al, 80 Fe	99.9(14,500)	569.8(82,700)	38.7	Mainly TF
DOP-4(0.3% W)-10	30 Th, 40 Al, 80 Fe, 16 Ni, 75 Rh	104.9(15,200)	568.6(82,400)	37.1	Mainly TF

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

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

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

tensile strength than for the undoped alloy. The ductility also increases with thorium content and reaches a peak value of 41.5% at a level of 200 ppm Th (DOP-14). The ductility drops considerably with more than 500 ppm Th in the alloy.

The fracture surfaces of tensile specimens were examined by optical microscopy, and the results are also presented in Table 1.1. Alloying with thorium apparently strengthens the grain boundary and suppresses the grain boundary fracture. The alloys doped with 100 and 200 ppm Th showed mainly transgranular fracture at 650°C, while the undoped alloy showed mainly grain-boundary separation. However, the alloys doped with 500 and 1000 ppm Th exhibited mixed fracture modes at 650°C, and the propensity for grain-boundary separation increases with thorium content. Thus, in terms of fracture behavior, the optimum amount of thorium in the Ir-0.3% W alloy should be around 200 ppm.

The microstructure of the thorium-doped alloys was examined metallographically after short-term heat treatments at 1500 and 1800°C. The microstructures are shown in Figs. 1.1–1.3. The heat-treated specimens show a recrystallized structure with second-phase particles, probably ThIr_5 , (Ref. 2) within the grains and on grain boundaries. The amount of second-phase particles increases with thorium content, and some particles actually line up into stringers in the 1000 ppm Th alloy [Figs. 1.1(d), 1.2(d), and 1.3(d)]. We believe this causes the low ductility of WTh-2 at 650°C (see Table 1.1). The grain growth resistance increases with thorium content, particularly after a complex heat treatment at 1800°C (Fig. 1.3). The refinement of grain structure is apparently due to the presence of second-phase particles which pin the grain boundary and retard its growth.

Characterization of impact behavior of Ir-0.3% W alloys indicates³ that both the grain size and fracture behavior at 650°C affect the impact properties of this alloy. Concern of both factors suggests that, in terms of impact properties alone, the optimum amount of thorium in Ir-0.3% W alloys should be somewhere between 200–500 ppm. The tensile impact properties of the thorium-doped alloys are reported in the "Physical and Mechanical Metallurgy of Heat-Source Containment Materials" chapter (chapter 2 of this report).

Fig. 1.1. Microstructures of Ir-0.3% W Alloys Doped with Various Levels of Thorium: (a) WTh-1, 50 ppm Th; (b) DOP-22, 100 ppm Th; (c) DOP-14, 200 ppm Th; (d) WTh-2, 1000 ppm Th. (Heat treatment for all alloys: 1 hr at 1500°C.) 100 $\times$.

Fig. 1.2. Microstructures of Ir-0.3% W Alloys Doped with Various Levels of Thorium: (a) WTh-1, 50 ppm Th; (b) DOP-22, 100 ppm Th; (c) DOP-14, 200 ppm Th; (d) WTh-2, 1000 ppm Th. (Heat treatment for all alloys: 19 hr at 1500°C.) 100 $\times$.

Fig. 1.3. Microstructures of Ir-0.3% W Alloys Doped with Various Levels of Thorium: (a) WTh-1, 50 ppm Th; (b) DOP-22, 100 ppm Th, (c) DOP-14, 200 ppm Th; (d) WTh-2, 1000 ppm Th. (Heat treatment for all alloys: 1 hr at 1500°C + 1 hr at 1800°C + 1 hr at 1500°C.) 100×.

To identify the important dopants in DOP-4, three new alloys (DOP-13, -15, and -16), with different combinations of aluminum and iron additions with thorium (dopant concentrations listed in Table 1.1), were prepared and fabricated into 0.76-mm- (0.30-in.-) thick sheets.³ The tensile properties of these doped alloys were determined at 650°C after a heat treatment of 1 hr at 1500°C. The results are presented in Table 1.1 and are compared with DOP-4(0.3% W)-10 and undoped WG. Doping with 40 ppm Al and/or 80 ppm Fe, in addition to 30 ppm Th, increases the yield and tensile strengths of the Ir-0.3% W alloys. Aluminum appears slightly more effective in strengthening. The alloys doped with DOP-4 series of dopants had tensile elongation in the range 37-40%. All the alloys doped with DOP-4 series of dopants showed transgranular fracture at 650°C. The tensile impact properties of these alloys are reported in chapter 2 of this report.

Figure 1.4 compares the microstructures of DOP-13 (30 ppm Th, 40 ppm Al) and DOP-15 (30 ppm Th, 80 ppm Fe) to rationalize the effect of aluminum and iron on grain structures. After a 1-hr anneal at 1500°C, the DOP-13 specimen showed distinctly elongated grain structure while DOP-15 exhibited, more or less, an equiaxed grain structure. In terms of number of grains across the specimen thickness, DOP-13 had 23.9 as compared to 15.6 for DOP-15. The DOP-13 alloy had a finer grain structure even after the 19-hr anneal at 1500°C [Fig. 1.4(c) and (d)]. Thus, doping with 40 ppm Al has distinct effects in the grain structure of the Ir-0.3% W alloy.

1.2.2 Long-Term Grain Growth Studies of Iridium Alloys — D. E. Harasyn

The results of the grain growth study on Ir-0.3% W alloys are shown in Figs. 1.5, 1.6, and 1.7. The study included three alloys (undoped WER and WG batches and the DOP-4 alloy) heated at 1310, 1415, and 1530°C for times to 2000 hr. These data show little difference in grain size between WG and WER for almost any given time and temperature. A spot check of WC and WE alloys (earlier PICS production material) shows little difference from WG and WER alloys. The grain size of DOP-4 is always smaller than WG and WER at all times and temperatures shown. The curves approach a slope of $-1/2$ at the larger grain sizes (i.e., longer time or higher temperature). Such behavior is expected for alloys that obey the ideal grain

Fig. 1.4. Microstructures of Doped Ir-0.3% W Alloys: (a) DOP-13 (30 ppm Th, 40 ppm Al), Heat Treatment of 1 hr at 1500°C; (b) DOP-15 (30 ppm Th, 80 ppm Fe), Heat Treatment of 1 hr at 1500°C; (c) DOP-13, Heat Treatment of 19 hr at 1500°C; and (d) DOP-15, Heat Treatment of 19 hr at 1500°C. 100 $\times$.

Fig. 1.5. Number of Grains Across a 0.64-mm- (0.025-in.-) Thick Ir-0.3% W Alloy Sheet vs Annealing Time at 1310°C.

Fig. 1.6. Number of Grains Across a 0.64-mm- (0.025-in.-) Thick Ir-0.3% W Alloy Sheet vs Annealing Time at 1415°C.

Fig. 1.7. Number of Grains Across a 0.64-mm- (0.025-in.-) Thick Ir-0.3% W Alloy Sheet vs Annealing Time at 1530°C.

growth law. We will try to fit the data to the grain growth law and, if possible, determine an activation energy.

The error bars shown in Figs. 1.5, 1.6, and 1.7 represent the standard deviation of the average number of grains across a 0.64-mm (0.025-in.) thickness. They are not a measure of the reproducibility of the data but simply an indication of how closely the average value could be determined for an individual sample.

We are continuing our long-term grain growth studies on the doped iridium alloys at 1330°C. The data for samples aged to 2000 hr are summarized in Table 1.2. The Ir-0.3% W alloys containing 200 and 1000 ppm Th are much more resistant to long-term grain growth, with 5 to 7 times more grain boundaries across the sheet thickness after 980 hr at 1330°C as compared to the undoped alloys, and 2 to 3 times that for the DOP-4 alloy after 2000 hr at 1330°C

Table 1.2. Grain Size Data for Ir-0.3% W Alloys
Heat Treated at 1330°C

Alloy Designation	Amount Thorium Added to Melt (ppm)	Number of Grain Boundaries Across a 0.64-mm (0.025-in.) Thickness					
		480 hr SD ^a		980 hr SD ^a		2000 hr SD ^a	
WER	0	7.8	0.4	6.3	0.4	—	—
WG	0	8.0	0.5	7.5	0.6	—	—
DOP-4	30	21.5	0.7	18.3	0.6	13.5	0.6
DOP-14	200	34.3	0.5	31.5	0.8	30.3	1.0
WTh-2	1000	49.7	0.7	44.4	1.1	42.2	1.0

^aStandard deviation of the average, based upon 12 measurements.

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

Previous work in advanced iridium alloys indicates that Ir-2% Ru, Ir-1.92% W, and Ir-0.3-0.6% Hf, have the most promising properties in terms of oxidation resistance, tensile properties, recrystallization, and grain growth behavior. However, our tensile tests reveal that alloying iridium with these elements does not improve grain boundary strength or suppress grain-boundary separation. In an attempt to further improve their mechanical properties, we have added dopants¹ to these alloys.

Two doped, advanced alloys were prepared by arc melting and drop casting into $19.05 \times 19.05 \times 38.10$ – 50.80 mm ($3/4 \times 3/4 \times 1\ 1/2$ – 2 in.) ingots. One of the alloys is Ir–2% Ru–0.3% W doped with 200 ppm Th (DOP-17); the other is Ir–2% Ru–0.5% W doped with DOP-4 dopants (DOP-18). The starting material for these ingots was electron-beam melted and the dopants were added as arc-melted master alloys. Because of some difficulty in casting the DOP-18 alloy, it took 4 attempts at drop casting to get a sound ingot. However, there was no difficulty in drop casting the DOP-17 alloy. The ingots were then clad in molybdenum and fabricated into 0.76-mm- (0.030-in.-) thick sheets between 1200 and 1100°C. The evaluation of their properties is in progress.

1.3 Pt-Rh-W Alloys

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

Fabrication of Pt-3008 (Pt–30% Rh–8% W) sheets for LASL and ORNL qualification studies continues. Progress during March includes the following completed operations: (1) shipment to LASL experimenters of 2.28 kg (5.02 lb) of cold-rolled sheets for both impact capsule [1.39 kg (3.06 lb)] and compatibility [0.89 kg (1.96 lb)] test matrices; (2) delivery to ORNL experimenters of 1.38 kg (3.04 lb) of cold-rolled sheets for both mechanical property [0.92 kg (2.03 lb)] and welding [0.46 kg (1.01 lb)] characterizations; (3) fabrication of a second consumable vacuum-arc-melted scale-up ingot, which has been machined into a right circular cylinder of 73.7-mm- (2.90-in.-) diam $\times$ 111.8-mm- (4.40-in.-) height of 8.35-kg- (18.4-lb-) weight; and (4) issuance of a request for a commercial company to cast one argon-induction-melted slab [19-mm- (0.75-in.-) thick $\times$ 101.6-mm- (4.0-in.-) wide $\times$ 152.4-mm- (6.0-in.-) long] of about 5.61 kg (12.37 lb) from ORNL's prealloyed material. From the above ingot and slab, we plan to provide additional sheets of Pt-3008 for both LASL and ORNL studies.

Our fabrication of the Pt-3008 sheets that were provided to LASL and ORNL experimenters during March entails many distinct operations and parameters. Their history includes preparation of our first consumable vacuum-arc-melted scale-up ingot [5.9 kg (13.0 lb)]; extrusion of the ingot at

1300°C (2372°F) with a reduction of area of about 80% [usable material: 5.0 kg (11.0 lb)]; cross rolling of saw-cut sections of the extruded bar at 1200°C (2192°F) to a reduction in thickness of about 60% followed by recrystallizing at the rolling temperature; long rolling of the cross-rolled and annealed sections at 1000°C (1832°F) to a reduction in thickness of about either 83.5% or 76.7% to obtain 0.89- or 1.27-mm- (35- or 50-mil) thick hot-rolled plates, respectively; shearing in half the hot-rolled plates to facilitate edge trimming, stress relieving of the trimmed plates for 1 hr at 900°C (1652°F) and less than 1.3×10^{-2} Pa ($< 1 \times 10^{-4}$ torr); and rolling at room temperature to a reduction in thickness of about either 50 or 40% to obtain, respectively, cold-rolled sheets of 0.51- or 0.64-mm (20- or 25-mil) thickness. Losses of material from sawing and/or trimming of the ingot, extruded bar, hot-rolled plate, and cold-rolled sheets and from allocations for chemical analyses, rolling studies, and heat treatment studies account for 33% of the starting ingot weight.

Ultrasonic inspection of the cold-rolled sheets from our first scale-up ingot revealed two types of defects that slightly reduced the yield of "acceptable" material from 67 to 62%. After the stress-relieving treatment and before cold rolling, the particular sheet that came from the near vicinity of both the bottom of the ingot and the nose of the extrusion exhibited a blister of surface dimensions of 20-mm- (3/4-in.-) width $\times$ 50-mm- (2-in.-) length. After cold rolling to a reduction in thickness of about 40%, the blistering region lacked visual distinguishable features on both top and bottom surfaces of the sheet, but was readily defined with ultrasonic through transmission techniques. This defect could conceivably arise from internal surface-connected porosity ("cold shuts") in the ingot that entraps air during extrusion. Subsequent ingots will be machined to expose defects in the near vicinity of their outer surfaces, which can then be repaired by electron-beam-welding techniques before extrusion.

The second type of defect was associated with the shearing operation of the hot-rolled plates to facilitate edge trimming. After cold rolling, ultrasonic inspection showed indications of delaminations of various length-wise extents in the vicinity of the end of each sheet that was sheared in half at a thickness of about 1.27 mm (50 mils). Hot-rolled plates that were divided similarly into two pieces at a thickness of about 0.89 mm (35 mils) were free of this type of defect.

Based on ORNL's tentative compositional limits of 28.0–30.0 wt % Rh and 7.8–8.2 wt % W for Pt-3008, the rolled sheets from the extremes of the extruded bar contain 2–4% excess Rh and a slight deficiency (0.1 wt %) of tungsten, while a deficit of 0.7 wt % in both Rh and W alloying elements exists in the central areas of the extrusion. Table 1.3 presents the results of chemical analyses from specimens of cold-rolled sheets and permits comparison of these values to those obtained previously in corresponding regions of the nose, center, and tail of the extruded bar. Impurities that were detected at concentrations of less than 10 ppm and not included in Table 1.3 are B, Ca, Co, Au, In, Mn, Os, P, Ru, Ta, Th, U, and V. In

Table 1.3. Results of Chemical Analysis of Specimens from Extruded Bar and Cold-Rolled Sheets of Pt-3008 Alloy

Element	Detected Concentration					
	Extruded Bar			Cold-Rolled Sheets		
	Nose	Center	Tail	Nose	Center	Tail
<u>Alloying Elements, wt %</u>						
Rhodium ^a	30.40	30.70	30.30	33.76	27.29	32.45
Tungsten ^a	7.84	7.79	7.77	7.66	7.13	7.77
Platinum ^b	61.90	61.50	62.00	58.58	65.58	59.78
<u>Trace Elements,^c ppm</u>						
Aluminum	1	1	1	5	3	30
Carbon	21	17	35	68	56	53
Chromium	3	10	30	25	50	70
Copper	50	50	50	100	40	500
Iridium	25	20	15	10	15	12
Iron	50	50	100	70	70	100
Molybdenum	20	10	100	50	30	50
Nickel				30	20	40
Oxygen	9	8	10	18	24	29
Palladium				10	10	15
Silicon	10	10	3	20	30	100
Silver				10	10	15
Sulfur	5	1	5	15	7	100
Titanium				3	40	2
Zirconium	10	15	5	≤25	≤15	≤10

^aAtomic Absorption Method.

^bBy difference.

^cSpark source mass spectroscopy except carbon (by LECO combustion) and oxygen (by vacuum fusion).

general, the levels of trace elements in the rolled sheets from the three sampling locations are higher than the values for the extruded bar by up to one order of magnitude. These analyses indicate that the rolled sheets are of sufficient uniformity and purity and will serve adequately for forming and compatibility studies and for evaluation of properties.

Figure 1.8 shows general changes in microstructure and microhardness that occur during the fabrication of Pt-3008 sheets from a consumable electrode arc-melted ingot. As shown in Fig. 1.8, the cast alloy exhibits fairly equiaxed grains of average hardness of 200 DPH. This structure breaks up during extrusion to produce alternating layers of partially recrystallized and wrought appearing grains of average hardness of 285 DPH. During subsequent hot-rolling and annealing operations, the materials undergo complete recrystallization and grain growth. At this stage of fabrication, its appearance is again equiaxed grains of considerably smaller size than those of the cast ingot but of similar hardness (210 vs 200 DPH). Our warm-rolling and stress-relieving steps yield the desired fibrous structure and produce a hardness of 340 DPH. The final fabricating step, a 50% reduction in thickness at room temperature followed by a stress-relieving treatment, further raises the hardness of the sheet to 425 DPH.

The hardness of cold-rolled sheets of Pt-3008 increases with increasing amounts of reduction in thickness at room temperature. And, the rate of increase is greater for recrystallized than for fibrous-appearing material. Figure 1.9 shows the effect of various reductions on the hardness of specimens having different pre- or post-cold-rolling heat treatments. Before cold rolling, the material was fabricated as outlined in Fig. 1.8 and then heated 1 hr at either 900, 1000, or 1200°C (1652, 1832, or 2192°F) and less than 1.3×10^{-2} Pa ($< 1 \times 10^{-4}$ torr). If heated after cold rolling, similar time and pressure parameters were used for the final 870°C (1598°F) heat treatment. As shown in Fig. 1.9, the estimate of standard deviation of an individual hardness measurement is ± 9 DPH, and each plotted point represents the arithmetic mean of five replicated determinations from one specimen.

The microstructures of some of the hardness specimens for Fig. 1.9 that are in the warm-rolled and heat-treated, and cold-rolled and stress-relieved conditions are shown in Fig. 1.10. The warm-rolled materials that were heat treated at either 900 or 1000°C (1652 or 1832°F)

Y-136650

Consumable electrode arc-melted
200 DPH

Extruded
(1300°C, 80% reduction)
285 DPH

Hot-rolled (1200°C, 60% reduction)
and annealed (1200°C for 1 hr)
210 DPH

Warm-rolled (1000°C, 80% reduction)
and stress relieved (900°C for 1 hr)
340 DPH

Cold-rolled (20°C, 50% reduction)
and stress relieved (870°C for 1 hr)
425 DPH

0.010"

Fig. 1.8. Microstructure of Pt-3008 Sheet at Various Stages of Fabrication. Etched electrolytically. 100×.

Fig. 1.9. Effect of Rolling at Room Temperature on Diamond-Pyramid Hardness Number of Pt-3008 Sheets of Differing Heat Treatments.

WARM-ROLLING
TEMPERATURE

900°C
(1652°F)

1000°C
(1832°F)

1200°C
(2192°F)

As-WARM-ROLLED

MICRONS
100X
INCHES
700
600
500
400
300
200
100
0.025
0.020
0.015
0.010
0.005

50% COLD ROLLED AND STRESS RELIEVED
AT 870°C (1592°F)

Fig. 1.10. Polished and Etched Specimens of Pt-3008 Sheets Fabricated from Extruded Bar.

before cold rolling retained their fibrous structures throughout the various reductions at room temperature and subsequent stress relief at 870°C (1598°F). Their hardness values were, respectively, 461 ± 4 and 459 ± 11 DPH after cold rolling a total of 50% and 426 ± 11 and 436 ± 12 DPH after stress relieving.

The 1200°F (2192°F) treatment before cold rolling of these hardness specimens fully recrystallizes the warm-rolled structure, as shown in Fig. 1.10. During cold rolling, the fairly equiaxed grains distorted and elongated in the direction of rolling, but still exhibited granularity after a total reduction of 50% in thickness. For this amount of cold rolling, the stress-relieving treatment lowered its hardness from 431 ± 17 to 388 ± 13 DPH.

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

Preparation of gas tungsten-arc weld specimens from sheet LCPT-10 has been hindered by cracking which frequently occurs during the specimen punching operation. Results of tests on the specimens which have been successfully punched are given in Table 1.4, to be compared with previously reported data.⁴ Also included are base metal data for reference purposes. As noted previously, reannealing 1 hr at 1200°C causes an improvement in room-temperature ductility to 10% total elongation, and an increase in strength (specimen 10-106 vs specimen 10-107).

The longitudinal gas tungsten-arc specimen (10-17) shows strength and ductility essentially equivalent to base metal. Welds made by either process in reannealed sheet (10-18 and 10-19) show reduced strength and ductility when compared to reannealed base metal (10-107), with the gas tungsten-arc weld showing lower ultimate strength than the electron-beam weld. Fracture occurred in the gas tungsten-arc weld metal and outside the electron-beam weld metal.

Postweld heat treatment of 1 hr at 1200°C improves both the strength and ductility of welds made by either process. Again, though, the gas tungsten-arc weld has lower ultimate strength and only a marginal improvement in ductility, and fracture occurs in the weld metal.

Three additional specimens are being prepared by machining to assure that the data obtained from punched specimens are not seriously influenced by the punching operation.

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

Specimen Designation	Test Temperature (°C)	Type ^b	Strength, MPa(ksi)		Total Elongation (%)	Fracture Location
			0.2% Yield	Ultimate		
10-17	20	L-GTA	413.7(60)	537.8(78)	2.0	—
10-18	20	R-T-GTA	275.8(40)	399.9(58)	2.0	weld
10-19	20	R-T-EB	275.8(40)	482.6(70)	2.0	base metal
10-20	20	R-T-GTA-P	344.8(50)	441.3(64)	2.5	weld
10-21	20	R-T-EB-P	344.8(50)	496.4(72)	5.0	base metal
10-106	20	B	473.2(68.6)	500.2(72.5)	2.0	—
10-107	20	BR	511.7(74.2)	632.9(91.8)	10.0	—

^aFull-penetration welds in 0.56-mm (0.022-in.) sheet, previously annealed 1 hr at 1000°C.

^bB = base metal; L = weld longitudinal to specimen axis; T = weld transverse to specimen axis at center of gage length; R = base metal reannealed 1 hr at 1200°C prior to welding; GTA = gas tungsten-arc weld; EB = electron-beam weld; and P = postweld annealed 1 hr at 1200°C.

Two sheets (2B and 5A) from the scale-up heat E1 have been received, and electron-beam weld specimens are being prepared.

1.4 Ruthenium and Rhodium Alloys

H. Inouye

1.4.1 Ruthenium Fabrication and Characterization

The fabricability and properties of ruthenium- and rhodium-base alloys are being characterized in an attempt to develop suitable cladding materials for isotopic fuels with lower density and/or lower cost than iridium- or platinum-base alloys. As previously reported, the fabrication of cast ingots of ruthenium alloys using procedures suitable for iridium- or platinum-base alloy sheet resulted in severe ingot cracking.⁵

In order to establish a suitable rolling temperature, three electron-beam-melted buttons of ruthenium, weighing about 75 g each, were canned in molybdenum and reduced about 50% in thickness by rolling at 1300, 1400, and 1500°C, then further rolled at 1000°C to 2.032-mm- (0.080-in.-) thick sheet for a total reduction of about 80%. Although each sheet was free of visible cracks, numerous pinholes were present whose number and size decreased with rolling temperature.

A powder compact of ruthenium and a Ru-0.5% La alloy, sintered to about 90% of theoretical density, was also rolled to 1400°C in a molybdenum can from a thickness of 5.08 mm (0.20 in.) to about 2.54 mm (0.10 in.), then further rolled to 1.27-mm- (0.05-in.-) thick sheet at 1000°C. Both sheets also contained numerous pinholes. These rolling experiments indicate that the pinholes, being common to both the cast ingots and the sintered compacts, are caused by the molybdenum can and not attributable to the grain size or grain morphology of the ruthenium alloys.

Phase relationships in the ruthenium-rhodium and ruthenium-platinum alloy systems are being studied metallographically and by x-ray analyses. The solubility of rhodium in ruthenium and ruthenium in rhodium has been verified to be at least 20 at. % as reported.⁶ Four ruthenium-platinum alloys containing up to 20 at. % Pt have been cast, sectioned, and heat treated for phase studies.

1.5 References

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

2.1 Introduction

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

2.2 Characterization of Impact Properties

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

2.2.1.1 DOP-4 Dopant Effect. — Tensile specimens of DOP-13, -15, and -16 alloys were heat treated 19 hr at 1500°C and then impact tested at 1350°C at a velocity of 85 m/sec (280 fps) to determine the effects of DOP-4 series of dopants on impact properties. The results are presented in Table 2.1 and are compared with DOP-4 alloy and undoped WG. The alloys DOP-13, -16, and -4, all doped with 40 ppm Al (in addition to 30 ppm Th), had 26.6–27.2% elongation, 56–64% reduction of area, and their fracture mode was transgranular fracture and ductile rupture. In comparison, the DOP-15, not doped with aluminum, has only 15.1% elongation and 39% reduction of area, and its fracture mode is grain-boundary separation and transgranular fracture. These results strongly indicate that aluminum, in addition to thorium, has very significant effects on improving impact properties of Ir–0.3% W alloys.

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

Table 2.1. Effect of DOP-4 Series of Dopants on Tensile Impact Properties of Ir-0.3% W Alloys Heat Treated 19 hr at 1500°C and Tested at 85 m/sec (280 fps) at 1350°C

Specimen	Nominal Dopant Concentration (ppm)	Grain Size ^a	Elongation (%)	Reduction of Area (%)	Fracture Mode ^b
WG-202	None	5.8	10.7	24	Mainly GBS
DOP-15	80 Fe, 30 Th	8.1	15.1	39	TF and GBS
DOP-13	40 Al, 30 Th	12.2	27.2	64	TF and DR
DOP-16	40 Al, 80 Fe, 30 Th	10.3	26.6	56	TF and DR
DOP-4	40 Al, 80 Fe, 30 Th, 16 Ni, 75 Rh	9.9	26.8	60	TF and DR

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

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

2.2.1.2 Thorium Concentration Effect. — The tensile impact properties of Ir-0.3% W alloys doped to 1000 ppm Th were determined for three heat-treatment conditions: 1 hr at 1500°C, 19 hr at 1500°C, and a complex heat treatment of 1 hr at 1500°C plus 1 hr at 1800°C plus 1 hr at 1500°C. All the heat-treated specimens were impacted at a velocity of 85 m/sec (280 fps) at 1350°C; the results are presented in Table 2.2. After a heat treatment of 1 hr at 1500°C, the impact elongation increases with increasing thorium content in the alloy to 100 ppm, but becomes insensitive above this level. The alloys showed ductile rupture with more than 85% reduction of area after doped with 50 ppm or more. After a heat treatment of 19 hr at 1500°C, the impact elongation and reduction of area also increased with thorium content and became insensitive to thorium content between 200–1000 ppm. After a severe heat treatment between 1500 and 1800°C, DOP-14 and WTh-2 had 25.2–25.4% elongation and 71–73% reduction of area and fractured by ductile rupture and transgranular fracture, which are distinctly better than the alloys doped with lower amounts of thorium (see Table 2.2). Thus, the DOP-14 and WTh-2 are most resistant to degradation due to severe heat treatment. The impact data (in Table 2.2), in connection with microstructure analysis and fracture behavior in section 1.2.1, suggests that the Ir-0.3% W doped with thorium at a level of 200–500 ppm may have the best impact properties.

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

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

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

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

^cNot able to measure; gage section destroyed.

Figure 2.1 shows the plot of impact elongation and fracture behavior as a function of the number of grains across the 0.64-mm- (0.025-in.-) thick sheet for undoped WG, DOP-4, and DOP-14. For the same grain size, the impact properties of DOP-14 are no better than those of DOP-4. Thus, we believe that the superior impact properties of DOP-14 are due to the precipitation of significant amount of second-phase (probably ThIr₅) particles which retard grain growth during high-temperature heat treatments.

Fig. 2.1. Plot of Impact Elongation as a Function of Grain Size for Ir-0.3% W Alloys WG, DOP-4, and DOP-14.

2.2.1.3 Impact Temperature Effect. — Tensile specimens of Ir-0.3% W alloys were heat treated 1 hr at 1500°C, and then impact tested in the temperature range 950–1350°C to determine the effects of test temperature on impact properties of WG, DOP-4, -13, and -14. All the specimens were impacted at 85 m/sec (280 fps); the results are presented in Table 2.3 and Fig. 2.2 as a function of test temperature. The impact properties of WG and DOP-4 specimens are not sensitive to test temperatures above 1200°C; below this temperature, the elongation and reduction of area decrease with test temperature for these alloys. At 1050°C, the DOP-14 specimens had 29.2% elongation and 69% reduction of area, which are far superior to WG and DOP-4 at the same impact temperature. Thus, DOP-14 appears to be most resistant to low-temperature impact among these alloys. The WG specimen fractured mainly by grain-boundary separation and DOP-14 mainly by ductile rupture at these temperatures, while DOP-4 specimens changed fracture mode from ductile rupture above 1200°C to mixed grain-boundary separation and transgranular fracture at 1050°C. DOP-13 exhibited mainly grain-boundary separation at 950°C.

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

To study the effects of long-term heat treatments at the MHW fuel clad temperature on the physical and mechanical properties of Ir-0.3% W alloys, WG (undoped) and HD (doped with DOP-4 dopants) specimens were annealed at 1330°C for various times and then impacted at 1350°C and 85 m/sec (280 fps). The impact data are presented in Table 2.4 and plotted as a logarithmic function of the annealing time in Fig. 2.3. The impact elongation of both alloys decreases with annealing time, and this decrease is apparently due to grain growth at 1330°C (see the grain size data in Table 2.4). According to the plot in Fig. 2.3, WG material had a marginal ductility of 14% elongation after being annealed for only 30 hr at 1330°C. We believe Ir-0.3% W should have at least 14% elongation in order to survive the MHW/FSA-type impact. Extrapolation of DOP-4 data in Fig. 2.3 indicates that DOP-4 alloy may remain ductile after 10,000–100,000 hr of annealing at 1330°C. Thus, DOP-4 is much more resistant to degradation due to long-term heat treatment at 1330°C.

Table 2.3. Effect of Test Temperature on Tensile Impact Properties
of WG, DOP-4, DOP-13, and DOP-14 Sheet Specimens
Heat Treated 1 hr at 1500°C and
Tested at 85 m/sec (280 fps)

Test Temperature (°C)	Elongation (%)	Reduction of Area (%)	Fracture Mode ^a
<u>WG Specimens^b</u>			
1350	12.6	28	Mainly GBS
1250	13.5	27	Mainly GBS
1150	8.6	18	Mainly GBS
1050	5.4	13	Mainly GBS
950	4.5	12	Mainly GBS
<u>DOP-4 Specimens^c</u>			
1350	37.6	94	DR
1250	38.2	82	DR
1150	21.5	54	TF
1050	15.2	44	TF
1050	13.6	25	GBS and TF
<u>DOP-13 Specimen^d</u>			
950	10.3	24	Mainly GBS
<u>DOP-14 Specimens^e</u>			
1350	37.9	92	DR
1050	29.2	69	DR and TF

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

^bUndoped Ir-0.3% W.

^cIr-0.3% W doped with DOP-4 dopants.

^dIr-0.3% W doped with 30 ppm Th and 40 ppm Al.

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

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

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

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

^aUndoped Ir-0.3% W specimens.

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

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

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

^eAnneal 1 hr at 1300°C.

Fig. 2.3. Effect of Long-Term Annealing at 1330°C on Impact Elongation of WG and DOP-4 Specimens Impacted at 1350°C and 85 m/sec (280 fps).

2.3 Environmental and Impurity Effects

2.3.1 Pt-3008 Air Oxidation — H. Inouye

Oxidation tests of Pt-3008 tensile specimens in still air, scheduled for 10,000 hr, logged 3861 hr on March 22, 1976. As observed in a prior set of tests, a dark gray oxide film was present on the specimens oxidized at 800°C, but was not evident on specimens oxidized at 600 or 1000°C. Weight changes also confirm prior results as follows: (a) weight changes are very small at all test temperatures; (b) at 800°C, the specimens gain weight but lose weight at 1000°C; (c) weight changes at 600°C are too low to determine a trend.

Table 2.5 shows the effects of oxidation on the tensile properties at the oxidation temperature. No significant environmental effects were noted in specimens exposed to 3042 hr at 600°C; however, the data show a slight

Table 2.5. Tensile Properties of Pt-3008 (Heat Pt-5C)
Oxidized in Still Air^a

Exposure Conditions			Strength, MPa(ksi)		Elongation (%)	Reduction of Area (%)
Time (hr)	Temperature (°C)	Environment	Ultimate	0.2% Yield		
<u>600°C</u>						
1	1200	Vac (Annealed)	594.6(86.3)	198.4(28.8)	34.8	~100
1000	600	Vac (Control)	642.1(93.2)	232.9(34.8)	34.8	79.2
1000	600	Air	627.0(91.0)	231.5(33.6)	32.0	84.9
3042	600	Air	635.3(92.2)	248.7(36.1)	28.4	79.0
<u>800°C</u>						
1	1200	Vac (Annealed)	540.2(78.4)	182.6(26.5)	28.5	77.7
1000	800	Vac (Control)	496.8(72.1)	184.0(26.7)	27.0	63.8
1000	800	Air	347.3(50.4)	190.2(27.6)	18.2	21.9
3042	800	Air	184.0(26.7)	184.0(26.7)	1.6	2.7
<u>1000°C</u>						
1	1200	Vac (Annealed)	311.4(45.2)	170.2(24.7)	13.2	11.6
1000	1000	Vac (Control)	351.4(51.0)	134.4(19.5)	38.6	48.0
3042	1000	Vac (Control)	323.1(46.9)	109.6(15.9)	38.0	53.1
1000	1000	Air	188.1(27.3)	126.1(18.3)	8.8	14.0
3042	1000	Air	130.2(18.9)	101.3(14.7)	4.2	5.0

^aSpecimen gage dimensions: 12.7 × 3.175 × 0.64 mm (0.50 × 0.125 × 0.025 in.); strain rate: 0.2/min.

but progressive decrease in the ductility with exposure time to air. The higher strength of the control specimen relative to the annealed specimen is suggestive of an aging reaction at 600°C. Fig. 2.4(a) shows the microstructure of the fractured end of a specimen oxidized 1000 hr and tested at 600°C, and confirms the absence of any embrittlement.

Oxidation of the alloy at 800°C causes a reduction in the ductility at that temperature from 27 to 18.2% elongation after 1000 hr and to 1.6% elongation after the 3042-hr exposure. Fig. 2.4(b) shows an embrittled surface zone and a ductile core after the 1000-hr exposure. Embrittlement also occurs in the specimens oxidized at 1000°C. At this temperature, the ductility decreases from about 38 to 8.8% after a 1000-hr exposure [see Fig. 2.4(c)] and is 4.2% after 3042 hr. It is noted that the ductility of the control specimens at 1000°C has increased by a factor of 3 over the annealed specimen after aging. Therefore, the ductility minimum observed in annealed specimens has either shifted from its characteristic 1000–1100°C range or no longer exists after the thermal aging treatment in vacuum.

2.3.2 Pt-3008/ZrO₂ Compatibility — H. Inouye

Zirconia is often considered as a component inside of or as a structural support for noble metal capsules. Since there was some concern that the noble metal/oxide interactions observed in gettered inert gases, hydrogen, and ultrahigh vacuum^{1,2} might also occur between Pt-3008 and ZrO₂ in a heat-source environment, compatibility tests for this combination of materials were conducted in both helium and in vacuum.

Table 2.6 summarizes the tensile properties of Pt-3008 specimens in the aged condition (control) and after exposure to ZrO₂ in vacuum and to ZrO₂ in helium. The properties of specimens annealed 1 hr at 1200°C are also listed to show the effects of the thermal aging treatment. The data show the following:

(a) Thermal Aging Effects — For all test temperatures, the strength of the control specimens is consistently lower than the annealed specimens. Aging had no significant effect on either the elongation or the reduction of area, except at 1316°C. At this temperature, aging embrittles the alloy.

Fig. 2.4. Fractured Ends of Pt-3008 Tensile Specimens Oxidized 1000 hr in Air and Tested at (a) 600°C with 32% Elongation, (b) 800°C with 18.2% Elongation, and (c) 1000°C with 8.8% Elongation. 150 $\times$.

Table 2.6. Tensile Properties of Pt-3008 (Heat LCPT-4)
Vacuum Aged and Heated in Contact with ZrO₂^a

Exposure Conditions			Strength, MPa(ksi)		Elongation (%)	Reduction of Area (%)
Time (hr)	Temperature (°C)	Environment	Ultimate	0.2% Yield		
<u>25°C</u>						
1	1200	Vac (Annealed)	620.1(90.0)	361.0(52.4)	11.2	12.5
1007	1050	Vac (Control) ^b	511.9(74.3)	292.8(42.5)	10.4	d
1004	1050	ZrO ₂ -Vac ^b	618.0(89.7)	336.2(48.8)	12.6	10.5
1000	1050	ZrO ₂ -He ^c	344.5(50.0)	240.5(34.9)	6.8	9.6
<u>760°C</u>						
1	1200	Vac (Annealed)	567.1(82.3)	214.3(31.1)	27.4	85.1
1007	1050	Vac (Control)	536.7(77.9)	146.1(21.2)	36.8	83.6
1004	1050	ZrO ₂ -Vac	567.1(82.3)	199.1(28.9)	35.4	90.2
1000	1050	ZrO ₂ -He	421.7(61.2)	107.5(15.6)	39.6	88.7
<u>1093°C</u>						
1	1200	Vac (Annealed)	285.9(41.5)	158.5(23.0)	17.2	19.0
1007	1050	Vac (Control)	246.0(35.7)	112.3(16.3)	12.0	19.9
1004	1050	ZrO ₂ -Vac	259.1(37.6)	130.2(18.9)	24.0	27.4
1000	1050	ZrO ₂ -He	212.2(30.8)	68.2(9.9)	28.8	26.1
<u>1316°C</u>						
1	1200	Vac (Annealed)	150.2(21.8)	121.3(17.6)	40.0	44.2
1007	1050	Vac (Control)	78.6(11.4)	67.5(9.8)	8.0	42.0
1004	1050	ZrO ₂ -Vac	143.3(20.8)	108.9(15.8)	41.2	49.5
1000	1050	ZrO ₂ -He	93.0(13.5)	60.6(8.8)	26.8	3.6

^aSpecimen gage dimensions: 12.7 × 3.175 × 0.64 mm (0.5 × 0.125 × 0.025 in.); strain rate: 0.2/min.

^bPressure = 1.3×10^{-6} Pa (1×10^{-8} torr).

^cPressure = 78 MPa (600 torr).

^dSpecimen failed in pinhole.

(b) ZrO₂/Vacuum Effects — The strength of the specimens exposed to ZrO₂ are always higher than the strength of the control specimens with no indication of embrittlement. The strength increase suggests that the noble metal/oxide reaction mentioned previously might have occurred or that ZrO₂ retards the thermal degradation effects discussed previously.

(c) ZrO₂/He Effects — Both the ultimate and, in particular, the yield strength of the specimens exposed to the ZrO₂/He environment are significantly lower than those of the control specimens, especially at 760 and 1000°C.

Figure 2.5 shows the microstructures resulting from the exposure of Pt-3008 to ZrO_2 in vacuum and in helium. A most surprising result is the extremely large grain size of the specimen exposed to the ZrO_2/He environment. This extensive grain growth is probably the reason for the large reduction in the yield strengths noted for this environment in Table 2.6. Because Fig. 2.5(b) shows no evidence of a Pt-3008/ ZrO_2 interaction, it appears that the higher strengths of these specimens exposed to this environment, as compared to the control specimens, are due a smaller grain size [compare Fig. 1.2(a) with Fig. 1.2(b) (p. 5 of this report) in the unstressed condition]. The specimens fail intergranularly in Fig. 1.2(a) and (c) at 1316°C . The unusual failure mode shown in Fig. 1.2(b) results from recrystallization at or during cooling of the specimen from the test temperature.

We conclude from these relatively short-term exposures that thermal aging degrades the slow-strain tensile properties of Pt-3008 and is aggravated when the effects of the ZrO_2/He environment are superimposed on the thermal effects. In contrast, the $\text{ZrO}_2/\text{vacuum}$ environment appears to counteract the thermal effects and, in a sense, stabilizes the annealed properties. Because the observed effects are best explained in terms of the grain size, it is concluded that grain boundary impurities play an important role in the property changes.

2.3.3 Pt-3008 Physical Properties — H. Inouye

Moduli and Poisson's ratio vs temperature were reported previously.³ Because a value of about 0.17 for Poisson's ratio was abnormally low for metals, the measurements were repeated on a new wire specimen [2.03-mm- (0.08-in.-) diam $\times$ 101.6-mm- (4-in.-) long]. The new values are plotted as a function of temperature in Figs. 2.6, 2.7, and 2.8. These data were obtained by the sonic method, as previously done, through the efforts of J. P. Hammond of the Metals and Ceramics Division, ORNL.

UNSTRESSED CONDITION

FRACTURE ENDS

UNSTRESSED CONDITION

FRACTURE ENDS

Fig. 2.5. Microstructures of Pt-3008 Aged 1000 hr at 1050°C and Tensile Tested at 1316°C. (a) Control specimen; (b) ZrO₂/vacuum; and (c) ZrO₂/helium. 100×.

Fig. 2.6. Young's Modulus (E) of Pt-3008 as a Function of Temperature.

Fig. 2.7. Shear Modulus (G) of Pt-3008 as a Function of Temperature.

Fig. 2.8. Poisson's Ratio (ν) of Pt-3008 as a Function of Temperature.

2.4 Microanalysis Studies

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

2.4.1 The Role of Thorium in Ir-0.3% W Alloys

The improvements in high-temperature impact performance, obtained by doping Ir-0.3% W alloys with the DOP-4 dopants, has given rise to questions concerning the relative importance of the five dopants (Al, Fe, Ni, Rh, Th) and the mechanism(s) whereby they induce these improvements. Analysis of doped and undoped Ir-0.3% W alloys, using Auger electron spectroscopy, have consistently indicated that thorium is the only one of the DOP-4 dopants that segregates to the grain boundaries at detectable levels.⁴⁻⁷

Recent impact test results on Ir-0.3% W alloys doped only with thorium showed that, except for very large grain-size samples, these alloys fail in a ductile mode with reductions of area of approximately 90%. This suggests that thorium, by itself, alters the mechanical properties of the grain boundaries so that they no longer limit the high-temperature impact properties of these alloys.

Since thorium seems to be the element that directly influences the mechanical properties of the grain boundaries, a detailed study of the mechanism by which it affects these properties has been initiated.

The variation in properties with thorium content have been studied using alloys having nominal thorium concentrations between 30 ppm (DOP-4) and 1000 ppm (WTh-2) using AES and conventional metallographic techniques. The WTh-2 alloy contains a significant fraction of second phase (presumably Ir₅Th), suggesting that the solubility of thorium in Ir-0.3% W is certainly less (probably much less) than 1000 ppm. Comparison of microstructures in alloys containing various amounts of thorium suggests that the thorium solubility in Ir-0.3% W may be less than 30 ppm.⁸

The thorium concentration on intergranular fracture surfaces has been determined using AES for the alloys indicated in Table 2.7. The procedure for AES analysis has been described previously;⁹ however, the analysis of alloys containing 1000 ppm Th (WTh-2) required a somewhat different procedure and analysis of the results. Initial AES studies of heat WTh-2 indicated approximately the same or slightly lower thorium concentrations

Table 2.7. Alloys Examined Using
Auger Electron Spectroscopy

Heat	Thorium Doping Level
WC-71	None
WCR-93	
WE-74	
WER-113	
WG-200	
WG-202	30 ppm
DOP-4 (Ir-0.3% W) -1	
DOP-4 (Ir-0.3% W) -4	
DOP-4 (Ir-0.3% W) -7	
DOP-4 (Ir-0.3% W) -10	50 ppm
WTh-1	
DOP-14	
WTh-2	
	200 ppm
	1000 ppm

on fresh fracture surfaces as compared to alloys containing lower thorium additions (DOP-4 alloys). It was also observed that, following fracture in the ultra-high vacuum (UHV) system, the oxygen AES signal increased with time, and the thorium AES signal decreased with time. The changes in the iridium signal were minimal, and the mechanism(s) responsible for these changes in oxygen and thorium signals as a function of time have not been explored. It seems reasonable, however, to suggest that the increase in oxygen signal is the result of oxygen absorption on the fracture surface. The reason for the rather larger effect of this oxygen absorption on the thorium signal than on the iridium signal is not clear. The oxygen absorption occurs in spite of vacuum system base pressures in the range of 1.3×10^{-8} Pa (1×10^{-10} torr) and the use of a liquid-nitrogen-cooled titanium sublimation pump to bring the system pressure into the range of 1.3×10^{-9} Pa (1×10^{-11} torr) while the sample is being analyzed. Thorium is, of course, a highly active element with a strong affinity for oxygen, and, hence, thorium-rich fracture surfaces might be expected to interact with the residual oxygen in the AES vacuum system more strongly than surfaces not enriched with thorium.

In order to obtain a valid indication of the thorium signal at the moment of fracture, energies between 50–75 eV were scanned repeatedly

following fracture of a sample of WTh-2. Starting a little more than one minute after the sample was fractured, the 54-eV iridium peak and the 67-eV thorium peak were recorded at 9- and 10-sec intervals. Approximately 7 min after the sample was fractured, the scan rate was reduced to allow the peaks to be recorded at 40-sec intervals. Figure 2.9 shows the results of these measurements for several time intervals up to 30 min after the sample was fractured. The decrease in the thorium peak height relative to the iridium peak height is easily observed. The thorium peak height is plotted as a function of time (after fracture) in Fig. 2.10. The heights of individual peaks are represented as dots in Fig. 2.10 while crosses indicate the average thorium peak height over a 2-min interval centered about the point, and squares indicate the average thorium peak height in a 10-min interval centered about the point. Extrapolation of the data in Fig. 2.10 to the moment of fracture ($t = 0$) suggests a thorium peak height in the neighborhood of 14 mm (1/2 in.). The iridium (54 eV) peak height at the moment of fracture is estimated to be about 153 mm (6 in.), yielding a ratio of thorium (67 eV) to iridium (54 eV) peak heights (R) of about 0.0915. Figure 2.11 shows the variation of R with the thorium doping level for alloys studied thus far. The right axis in Fig. 2.11 also indicates the estimated atomic percent thorium to be associated with each point.* While there is some scatter in the data, and while uncertainty remains about how much of the thorium that is added to the alloys is retained after melting, the data suggests that for doping in the range 30–1000 ppm Th, the concentration of thorium at the grain boundaries does not change significantly.

A series of low thorium (5–25 ppm) Ir–0.3% W alloys is being prepared in order to study the effect of bulk thorium content on high-temperature impact properties and grain boundary chemistry. We will be particularly interested in determining the minimum doping level of thorium that will result in acceptable high-temperature impact properties for alloys of any given grain size. Since we know that significant changes in grain boundary chemistry and impact properties occur with the addition of 30 ppm Th, the

*The atomic percent thorium indicated in Fig. 2.10 is estimated by assuming that our analysis is approximately 4.4 times as sensitive to the thorium as iridium and ignoring other elements on the fracture surfaces.

Fig. 2.9. Partial Auger Spectra of WTh-2 Showing the Iridium (54 eV) and Thorium (67 eV) Peaks as a Function of Time. Several time intervals up to 30 min (1800 sec) are shown.

Fig. 2.10. Variation in the Thorium (67 eV) Peak Height as a Function of Time after Fracture.

Fig. 2.11. Variation in Grain Boundary Thorium Concentration (Determined by Auger Electron Spectroscopy) as a Function of the Bulk Thorium Doping Level.

study of these low-thorium alloys may give insight into the mechanism(s) responsible for these changes. Additional understanding of these mechanisms could result in the selection of better dopants or dopant combinations.

Samples of heat WTh-2 (Ir-0.3% W-0.1% Th) have been examined by W. H. Christie* using ion microprobe mass analysis (IMMA). This technique has the advantage of being able to detect very small concentrations (in the ppm range) of certain elements in areas as small as a few micrometers. The sample of WTh-2 had a 1 hr at 1500°C heat treatment and was prepared for metallographic examination. The metallographic sample was then analyzed using IMMA. The mass spectrum of the sample indicated the presence of Ir, W, Th, and U, with the uranium concentration about one-twentieth that of thorium.

Rastering the ion beam to allow mapping of the various elements over the surface indicated the thorium to be concentrated in the small precipitates that are easily seen in WTh-2. There was some indication that the precipitates are somewhat richer in tungsten than the matrix material; however, more work will be necessary in order to establish this with confidence.

The sample of WTh-2 is being reground and repolished so that IMMA can be obtained on a sample with a surface that has not been etched, in case the etching removes the material of interest in the analysis.

2.4.2 Phosphorus Contamination and Embrittlement of Grain Boundaries in Ir-0.3% W Alloys

Evidence has been reported previously⁸ that indicates phosphorus contamination of the Ir-0.3% W cladding material may be occurring in single sphere FSA impacts (using a thorium fuel simulant). Limited evidence is also available⁸ that suggests that phosphorus contamination could lead to serious degradation of the high-temperature impact performances of these alloys.

Material from fuel sphere MHT-45 (made from EI lot 86 iridium) has recently been examined using AES, and the results appear to be consistent with those reported by others.¹⁰ A fresh intergranular fracture surface appeared to have about 5 at. % P on it, and sputtering less than 10 atom layers using Ar⁺ removed all traces of the phosphorus.

*Analytical Chemistry Division at ORNL.

Methods of contaminating Ir-0.3% W alloys with controlled amounts of phosphorus are currently being evaluated in order to provide material for studying the effect of phosphorus on the impact properties and grain boundary chemistry of these alloys.

2.4.3 Grain Boundary Analysis of Pt-3008 Alloys

Samples of Pt-3008 alloy from heats LCPT-32-4, LCPT-9-6, and LCPT-4-3 have been analyzed using Auger electron spectroscopy. Heat LCPT-32-4 has been reported to have good ductility (elongation is 40.2%) at 1093°C, while LCPT-9-6 and LCPT-4-3 have been reported to have significantly lower ductilities (elongations of 15 and 17%, respectively). AES analysis of these alloys indicated that heat LCPT-9-6 had a grain-boundary phosphorus contamination of about 1.5 at. % while heats LCPT-32-4 and LCPT-4-3 had about 0.5 at. % P at the grain boundaries. In addition to the phosphorus, each of these alloys had small, but detectable concentrations of silicon on them. LCPT-32-4 and LCPT-9-6 had approximately 2 at. % Si at the grain boundaries, while LCPT-4-3 had about half that amount. Inert ion sputtering experiments indicated that both phosphorus and silicon were preferentially segregated to the fracture surfaces.

2.5 Special Projects

2.5.1 Effects of Thermal Aging on the Mechanical Properties of Superalloys for Isotope Containment — J. P. Hammond

During the past quarter, the evaluation of the effects of long-term thermal aging (11,000 hr) at 816°C (1500°F) on the tensile properties of four isotopic containment superalloys (Haynes 25, Haynes 188, Hastelloy N, and Inconel 625) was completed. In the last quarterly progress report,¹¹ the results for the first three aforementioned materials was reported. The present report gives the tensile test results for Inconel 625, tensile test toughness values for all four alloys, the results of phase identification studies, and endeavors to explain the tensile test findings. The chemical analyses of the alloys and details of the methods of test were given in the last quarterly progress report.¹¹

2.5.1.1 Tensile Test Results on Inconel 625. — The tensile test results for Inconel 625 in the usual graphical form are given in Fig. 2.12. Results for two Haynes 25 alloys (0.1 and 0.36% Si), Haynes 188, and Hastelloy N, which were presented in the last quarterly,¹¹ showed long-term aging at 816°C not to have a very large effect on the tensile and yield strengths but to substantially influence the amount of total elongation. For room-temperature tests, elongations were depreciated in amounts from 74% (Hastelloy N) to 86% [Haynes 25 (0.36% Si)]. Figure 2.12 shows that aging Inconel 625 significantly lowers its yield strength for the lower test temperatures. Total elongation at room temperature again was significantly reduced from the solution-annealed value, but not to nearly so great an extent (33%) as for the other alloys. As in the case of the Haynes 25 and Haynes 188 alloys, a reversal in the extent of total elongation at the ductility minimum temperature was obtained for Inconel 625. Whereas at approximately 704°C (1300°F) Inconel 625 in the solution-annealed condition displays its lowest ductility (about 40%), an unexpected high value was obtained in this temperature region in the aged condition (Fig. 2.12). At still higher test temperatures, ductility increased to enormous values, which is characteristic solely of Inconel 625. Because ductility of all of the aged superalloys was quite high at approximately 760°C (1400°F), a temperature at which yield and tensile strengths had not begun to drop off very greatly, we surmised that the aged alloys would reflect high toughness values at this temperature. Consequently, the tensile data were analyzed to establish toughness response.

2.5.1.2 Tensile Toughness of Superalloys. — Toughness values were determined for the aged alloys at the ultimate and fracture tensile stresses by establishing areas under the engineering stress-strain graphs by the Johnson integration method. The results are presented in the form of bar charts in Fig. 2.13. The individual charts give energies to ultimate and fracture stress for tests successively at 24, 316, 760, and 982°C.

Significantly, in the case of all the aged cobalt-base alloys, highest toughnesses were obtained at 760°C (the temperature of the ductility minimum for the solution-annealed condition). These values proved 2 to 3 times higher than for the other test temperatures. Interestingly, the bar charts

Fig. 2.12. Comparisons Between Effects of Solution-Annealed and Aged Conditions on the Yield Strength, Ultimate Strength, and Total Elongation of Inconel 625.

Fig. 2.13. Energy of Deformation at Ultimate Tensile and Fracture Stress for Alloy Tested in Aged Condition. (Bar charts for individual alloys give average of two values at 24, 316, 760, and 982°C, consecutively.)

for the individual cobalt-base alloys are similar, reflecting similar metallurgy. The individual charts for the two nickel-base alloys (Hastelloy N and Inconel 625), on the other hand, are different. The toughness values obtained for Hastelloy N are comparatively low. Inconel 625 gave the highest toughnesses for tests at room temperature which also were higher by far than the room-temperature toughnesses of the other alloys.

The reversal in ductility response of aged cobalt-base alloys at the temperature of their ductility minimum was unexpected and was not previously reported. These findings suggest that overaged cobalt-base alloys and Inconel 625 may exhibit impressive toughness at temperatures around 760°C, as well as good strength and ductility. The aged Inconel 625 alloy should show like advantages at room temperature.

2.5.1.3 Fractography of Fracture Surfaces and Identification of Phases. —

The microstructures of the individual aged alloys at the location of fracture after tensile testing at 316°C were reported in the last quarterly progress report.¹¹ Additional metallographic examinations after tests at the other temperatures, together with electron-beam scans on the fracture surfaces, gave the following results.

Fractures on all of the aged superalloys tested at room temperature were intergranular in mode except in the cast of Inconel 625, which had a transgranular fracture. On the other hand, for the 760°C test temperature (location of ductility minimum for solution-annealed condition), the fractures of the aged superalloys were predominantly transgranular in character except in the case of Hastelloy N, which gave a definite intergranular failure. The type of fracture observed for the individual alloys at room temperature correlated directly with the microstructures. For example, Inconel 625 had a fine dispersion of disconnected precipitate platelets with little or no concentration of precipitates in grain boundaries. The cobalt-base alloys had brittle precipitates of a semi-continuous nature in grain boundaries, while Hastelloy N displayed a massive precipitate of more continuous character in grain boundaries.

The fracture results on specimens tested at 760°C could be only partially explained by the metallographic results and will be commented on further below. The results of the electron-beam scans corroborated the

metallographic findings. Scans on Inconel 625 which was fractured at room temperature showed highly ligamented topology, suggestive of ductility and transgranular mode of fracture and even more so for the fractures associated with the 760 and 892°C tests. The scans of fractures of the Haynes 25 alloys tested at room temperature indicated intergranular fracture mode and little or no ductility. On the other hand, the fracture surface associated with this aged alloy after testing at 760°C displayed the cone-in-cup feature, characteristic of ductile failure. The fracture mode was predominantly transgranular.

The phases that precipitated in the aged superalloys were separated from the shoulders of tensile specimens pulled at 316°C by electrolysis and identified by x-ray diffraction. The results are listed below in Table 2.8.

Table 2.8. Precipitates Formed in Superalloys at 816°C

<u>By Yukawa <i>et al.</i>¹²</u>		
Haynes 25: $M_7C_3 \rightarrow [M_{23}C_6] \rightarrow M_6C \rightarrow \text{Laves } Co_2W \rightarrow \mu Co_7W_6$		
<u>By Herchenroeder <i>et al.</i>¹³</u>		
Haynes 188: $[M_6C] \rightarrow M_{23}C_6 \rightarrow \text{Laves } Co_2W$		
<u>After 11,000 hr^a</u>		
Haynes 25, 0.36% Si: $\text{Laves } Co_2W(S)$; 4 lines ^b (S)		14.5 wt %
Haynes 25, 0.01% Si:		14.8 wt %
Haynes 188: $\text{Laves } Co_2W(VS)$; 2 lines ^b (M)		15.2 wt %
Hastelloy N: $(Mo, Ni)_{12} C$		5.0 wt %

^aDensity scale: VS, S, M, W, VW.

^bDid not correspond to any of the carbide phases previously reported.

The information at the top of the table shows the secondary phases that appear with increasing time of aging at around 816°C, according to previous investigations. The phases contained in the brackets are the primary carbide constituents incorporated in the alloys for retarding grain growth with metal working and heat treatment. The underlined phases contribute to strength at 816°C by coherency hardening. The data in the right-hand column at the bottom gives the amount of secondary constituent electrolytically separated from the test specimens based on total metal removed.

Observe from the data that the amount of precipitate formed with aging the individual cobalt-base alloys is about equal. This, together with the metallographic data, indicated that silicon level had little influence on the resulting aged microstructures. Judging from the relatively large amounts of the Laves Co_2W phase found in the aged Haynes 25 and Haynes 188 alloys, it appears that the aging process was well along after 11,000 hr at 816°C . Since this phase is reported to confer brittleness¹³ to structural alloys containing it, it is reasonable that the cobalt-base alloys tested at room temperature should display little ductility. The precipitate formed in aged Hastelloy N was shown to be $(\text{Mo,Ni})_{12}\text{C}$. This phase had not been previously reported in Hastelloy N.

Because calculations based upon simplifying assumptions indicated that the matrices of the aged cobalt-base alloys may have as much as half of their tungsten depleted as a result of forming tungsten containing precipitates, an electron probe analysis was made of the aged Haynes 25 alloy (0.36% Si). Using tungsten metal as a standard, repeated isolation probe analyses were conducted on the matrix of the aged specimen and compared to ones made on a like alloy in the solution-annealed condition. These tests indicated that 11,000-hr aging at 816°C depleted the matrix of approximately one-third of its tungsten content. Further, set scans made of the matrix of the aged alloy indicated even tungsten concentrations as opposed to a denuding effect, although this must be taken with caution since resolution of the probe beam was no better than about $5\text{ }\mu\text{m}$. Tungsten $\text{Mo}\alpha$ x-ray displays made of various fields of view in the aged alloy also failed to show denuding. Again, however, resolution was not high. Such tungsten x-ray displays conducted on this alloy in the solution-annealed condition revealed the primary carbide constituent of the alloy solution annealed to contain a substantial amount of tungsten (perhaps 50%). This was not expected.

2.5.1.4 Rationalization of Ductility and Toughness Results. — The results of the ductility curves for all of the aged superalloys are incorporated in a single illustration with information on mode of fracture with at room temperature, 760, and 982°C included therein in Fig. 2.14. Mode of fracture for the solution-annealed condition was taken from the work of Rhines and Wray.¹⁴ According to their generalized model on plastic

Fig. 2.14. Correlation of Ductility Response with Fracture Mode.

behavior, all ductile metals and solution-annealed superalloys should show a ductility minimum at elevated temperatures. They postulate at low temperatures, fracture occurs by the usual transgranular crack propagation mechanism, and ductility is high. At temperatures near the minimum, deformation occurs by grain boundary shear, and the intergranular voids formed

at triple points grow unhindered, causing intergranular fracture with drastic loss in ductility. At high temperatures, recrystallization occurs simultaneously with intergranular void formation, continuously breaking up the intergranular fracture path. As a result, ductility increases again.

Observe from Fig. 2.14 that overaging of the solution-annealed superalloys resulted in a reversal in the fracture mode as well as a complete inversion of the ductility response. Moreover, high ductility appears to equate with transgranular mode of failure, and low with intergranular. The transgranular mode of fracture for all alloys in both conditions at 982°C is indicated by the dashed enclosure because all alloys in the aged condition were not examined at this temperature. Whether or not an alloy at a very high temperature fails intergranularly would appear to depend upon the extent to which grain boundaries are weakened by proximity to melting temperature.

The fact that the aged superalloys gave high ductility and predominantly transgranular mode of fracture at the temperature of the ductility minimum was not expected. Since the test specimens, after all, contained ostensibly the same internal microstructures that gave brittle, intergranular fractures in these materials at room temperature, explanations of why are in order. Three factors appear to be involved.

Usually, the ductility minimum behavior in its most accentuated form is associated with age hardenable alloys in their solution-annealed condition. As the alloy is hot worked or pulled, a profuse precipitation of carbides occurs at grain boundaries and along deformation bands. In the case of the present overaged alloys, potential for precipitation of secondary phases in the temperature region in question was completely spent and so there was no opportunity for precipitation embrittlement. This helps explain why the overaged alloys failed to display the ductility minimum but not why these aged alloys display high ductility at 760°C as contrasted to the low at room temperature. It is likely that embrittling phases, such as Laves Co_2W , are not as brittle at 760°C as at room temperature. Lastly, the observed tungsten depletion effect that accompanied overaging is possibly involved. Because these particular cobalt-base alloys are dependent upon tungsten additive for their elevated-temperature strength via substitutional solute hardening, its depletion from the matrix could possibly result in improved elevated-temperature plasticity.

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3. ISOTOPE BRAYTON SYSTEM MATERIALS SUPPORT*

3.1 Introduction

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

3.2 C-103/Gaseous Interactions

H. Inouye and C. T. Liu

The helium-xenon environment within the closed-cycle loop of the BIPS and the vacuum environment external to it is expected to contain low levels of gaseous contaminants that could react with and degrade the mechanical properties of the structural components. The principal objectives of this study are (a) to determine as early as possible the feasibility of using C-103 as a prime structural component in BIPS environment, (b) to establish a data base to allow an estimate of the acceptable impurity limits for using C-103, and (c) to investigate methods to minimize or avoid the deleterious gas/metal reactions (e.g., gettering and substitution of less impurity-sensitive alloys).

*Progress on work performed under Activity No. KJ 30 01 02 2, 189a No. 00006.

The gaseous contaminants in the BIPS environment have not been identified; however, it is possible that the species and their concentration could approach that found in the helium coolant in gas-cooled reactors (Table 3.1). These gases are expected to be oxidizing and carburizing toward C-103 in the temperature range 700–1000°C. Previous work shows that T-111 is severely embrittled at test temperatures up to 1000°C by absorption of a few hundred ppm oxygen.¹

Table 3.1. Impurity Levels in Helium-Cooled Gas-Cooled Reactors² (in μ -atm)^a

Reactor	H ₂	H ₂ O	O ₂	CO	CO ₂	CH ₄
Dragon ^b	20	1	<1	10	0.4	3
Peach Bottom ^c	200	<10	—	10	—	15
NGT (est) ^d	20	<1	<1	10	<1	20

^aConversion: 1 μ -atm = 0.1 Pa (7.6×10^{-3} torr).

^bTypical analyses for system operating at 20 atm He. $t_{\max}$ (graphite) $\sim$ 1000°C, gas temperature, 700°C.

^cAverage analyses for system operating at 23 atm He. $t_{\max}$ (graphite) $\sim$ 950°C, gas temperature, 750°C.

^dNuclear Gas Turbine. General Electric Company estimate. For a system operating at 70 atm and a gas temperature of $\sim$ 815°C.

The effect of contaminating gases on the creep of T-111 tubing was determined using an existing environmental creep apparatus while components and specimens were being fabricated for testing of C-103 sheet specimens. The data given in Table 3.2 show that measurable strains occur when contaminants are present at pressures as low as 1.3×10^{-3} Pa (1×10^{-5} torr), but not at pressures of 1.3×10^{-6} Pa (1×10^{-8} torr). Figure 3.1 indicates that the absorption of oxygen by unstressed T-111 is accompanied by dilation of the same magnitude as shown in Table 3.2. Therefore, these preliminary data indicate that absorption of oxygen was primarily responsible for the observed strains and that creep contributed little, if any, to the overall strains of T-111. Because C-103 is expected to be chemically equivalent to T-111, the environmental effects discussed above for T-111 may also be applicable to C-103. Modification of the environmental creep

Table 3.2. The Effect of the Environment on the Creep of T-111^a at 1000°C and 15.2 MPa (2.2 ksi)

Strain (%)	Time to Indicated Strain (hr)		
	Vacuum (1.3×10^{-6} Pa)	Air (1.3×10^{-3} Pa)	CO (1.3×10^{-3} Pa)
0.1	b	14	1
0.2	b	55	2
0.3	b	133	22
0.4	b	228	222
0.5	b	338	450
0.6	b	456	>500

^aSpecimen: 2.03-mm- (0.08-in.-) OD $\times$ 0.25-mm- (0.01-in.-) wall tubing, annealed 1 hr at 1650°C.

^bNo measurable strain in vacuum. Time to 1% strain calculated to be 100,000,000 hr from Larson-Miller plot.³

Fig. 3.1. Correlation Between Oxygen Contamination and Dilation of Unstressed T-111 Tubing.

apparatus in which the above tests were conducted has been completed and testing of C-103 sheet specimens under conditions applicable to the BIPS is being initiated.

Specimens of C-103 have been exposed to 1.3×10^{-3} Pa (1×10^{-5} torr) CO at 1000°C for times to 2000 hr, and tensile tests and dilation measurements are in progress.

3.3 Alternate Alloys

H. Inouye and C. T. Liu

Stress-rupture and creep tests of molybdenum-rhenium alloys and Pt-3008 are planned for the next quarter to determine if they can be considered as alternate structural materials for C-103. Sheet specimens of Mo-33% Re, Mo-46% Re, and Pt-30% Rh-8% W have been fabricated for creep testing at 1000°C.

3.4 Welding and Brazing Evaluation

G. M. Goodwin

Review of the current status of critical components requiring welding and brazing development for the Isotope Brayton System was completed. The status of bimetallic joint development and testing was reviewed at NASA's Lewis Research Center on December 15, 1975. On the basis of metallographic examination and tensile tests of extensive screening samples and specimens aged 1000 hr at 1350°F, the direct braze of C-103 to Hastelloy X using the CM-50 alloy and the W-Ni-Fe graded transition joint brazed to C-103 with CM-50 and welded to Hastelloy X appears most promising. However, NASA has no plans for longer-term aging or microprobe analysis on these samples to determine the extent of interaction.

NASA has also fabricated excellent full-size bimetallic tube samples for thermal cycling tests using the CM-50 direct braze and transition joint processes using four different joint geometries. However, their planned thermal cycle tests may not be representative of thermal cycle these joints will experience in the Ground Demonstration System (GDS).

Also, the bimetallic joints were made using Nb-1% Zr pipe rather than C-103 because of availability. The possibility of hafnium intermetallic phases forming in the bimetallic joints with C-103 resulting in poor thermal cycle performance should be investigated.

The status of recuperator fabrication and testing was also reviewed. The current design modifications and less severe thermal ramp should minimize the internal leaks in the core and manifold splitter during thermal cycle testing of the prototype unit. There appear to be sufficient mechanical properties data on the Palniro 1 braze alloy used for the core. However, there is minimal elevated temperature data on the Palniro 7 braze alloy used for manifold-to-side plate braze. This braze is critical because it is a structural joint to contain the pressure of the working fluid and is a potential external leak path.

We have received the required materials and have initiated specimen preparation for bimetallic joint and recuperator braze alloy studies. Brazing alloys, including Coast Metals 50 (Ni-3.5% Si-2.5% B-1% Fe) in powder form, and Palniro 7 (Au-8% Pd-22% Ni) in wire and sheet form, have been procured from commercial sources. Tee-joint specimens are being prepared of Hastelloy X to Hastelloy X using Palniro 7 (recuperator application) and Hastelloy X to C-103 alloy with Coast Metals 50 (transition joint application). Lap-shear specimens will subsequently be prepared for short-term tensile tests and aging studies.

3.5 References

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4. SPACE NUCLEAR FLIGHT SYSTEMS HARDWARE

4.1 Introduction

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

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

D. E. Harasyn

4.2.1 Forming Disks

Production data for Ir-0.3% W forming disks from January through March 1976 are shown in Table 4.1. Microscopic delaminations in all metallographic samples from the lot HD 427-432 resulted in a nonconformance. Production was halted while an investigation into the cause of the delaminations was carried out. Although the cause has not been isolated, the microscopic delaminations did not affect the formability of a sample hemisphere hydroformed by Mound Laboratory nor did they result in unacceptable impact properties as discussed in section 4.3 of this report. Therefore, production has begun again as directed by ERDA-NRA. Our new schedule calls for shipment of 80 disks to MRC by the middle of May in order to complete the MJS mission. We foresee no scheduling problems in meeting the deadline, but a significant number of the 80 disks are expected to have the same type of surface delaminations as seen in HD 427-432.

*Progress on work performed under Activity No. KJ 30 01 03 2, 189a No. 00003.

Table 4.1. PICS Production Data

Ingot Number	Number Machined	Nonconformance Report ^{a,b}				Disks Shipped				Date Shipped	
		Disk Number									
		1	2	3	4	1	2	3	4		
HD-427	4			U		1	1		1	1/29/76	
HD-428	4	U	U					1	1	1/29/76	
HD-429	4					1	1	1	1	1/29/76	
HD-430	4					1	1	1	1	1/29/76	
HD-431	4			U	U	1	1			1/29/76	
HD-432	4		U			1		1	1	1/29/76	
TOTAL DISKS SHIPPED PER LOT:		18	CUMULATIVE TOTAL OF DISKS SHIPPED THIS QUARTER:								18

^aU = ultrasonic.

^bA metallography nonconformance applies to all disks made from HD 427-432 because of surface delaminations.

The surface delaminations are typically 0.1 mm (0.004 in.) in length and less than 0.01 mm (0.0004 in.) below the ground surface and usually cannot be seen at magnifications less than 200 $\times$. The largest delaminations are about 0.4-mm (0.16-in.) long. Figure 4.1 shows some typical delaminations found in longitudinal cross sections of test disks taken from HD 427-432. A spot check made on the next lot of sheets, HD 433-438, also revealed surface delaminations. The delaminations all run parallel to the wrought, fibrous structure of the metal. However, optical microscopy cannot give us any details of the fractured surface such as the presence of second phase particles and intergranular or transgranular fracture. In the production samples shown in Fig. 4.1, there are roughly 5-10 defects per centimeter of sample. Experimental samples prepared after HD 427-432 do not contain as many defects as severe as those in Fig. 4.1.

Metallographic examination is the only method we have so far of confirming the presence of the surface delaminations. The defects are too small to be detected by dye-penetrant, ultrasonic, or visual examination of the as-ground surface with or without the aid of optics. Apparent defects in the ground surface of HD-428, #1 (an ultrasonic reject) have been found by using scanning electron microscopy (see Fig. 4.2). However, we have not yet been able to correlate defects in the ground surface with the surface delaminations shown in Fig. 4.1. Moreover, there is no data base against which we can compare the photos in Fig. 4.2.

In order to determine the cause of the surface delaminations, we first have to determine at which point in the production process they first appear. The most probable processes which initiate the defects are (1) surface grinding, (2) sample cutting, and (3) metallographic preparation. Rolling and casting have been discounted because surface delaminations have not been detected in as-rolled sheet samples taken from the lots in question.

Y-137328

HD 427 P

HD 428 P

HD 429 P

HD 430 P

HD 431 P

HD 432 P

MICRONS
50
100
200
300 X
INCHES
0.002
0.004
0.008
0.010

Fig. 4.1. Surface Delamination in Metallography Samples of HD 427-432.

Fig. 4.2. Scanning Electron Micrographs of Ground Surface of the HD-428, #1 Forming Disk.

There are many possible causes for the surface delaminations. The most likely and most frequently mentioned causes are listed below.

Impurities in Powder	}	Material
Contamination of Ingots		
Loss of Dopants		
Melting and Casting	}	Procedure
Rolling		
Annealing		
Grinding		
Sampling		
Mounting/Grinding/Polishing		
Rolls	}	Equipment
Grinding Machine		
Grinding Wheel		
Cutoff Saw		
Metallographic Materials		

The list of possible causes can be grouped into three headings: material, procedure, and equipment. In addition, statistical fluctuations and crack propagation must be considered. A crack initiated by bad procedure or equipment at one point in the fabrication process can propagate in later fabrication steps.

Some preliminary experiments were done in an attempt to find a solution to the surface delamination problem without upsetting the schedule. Higher stress-relief temperatures and a new grinding wheel did not solve the problem. We don't believe that these results can be fairly interpreted at this time because the grinding specimens used already contained surface delaminations which may have propagated during continued grinding or in the metallographic process.

In order to reduce the number of variables, an experiment was done to look at the material alone. Pieces were removed from the unground skeletons of HD-406 and -428. HD-406 was from the second lot of HD material and had been fabricated successfully without any indication of surface delaminations. The pieces were simultaneously surface ground, sampled, and prepared for metallographic examination. Each metallographic sample was about four times the size of typical production samples in order to reduce the chances of being misled by statistical fluctuations. HD-406 had three surface delaminations while HD-428 had five. So, material that had been successfully fabricated 6 months earlier now contains the same defects seen in HD 427-432 when it is fabricated today. This is a strong indication that any chemical or metallurgical difference between the lot HD 427-432 and previous lots is not the primary cause of the surface delaminations. Weaker supporting evidence is that the trace impurity levels in HD 427-432 are not significantly different from previous lots of HD material. Also, as seen by optical microscopy, there is no microstructural difference between HD 427-432 and previous lots.

More experiments are needed to eliminate the variables that have been listed. We had originally believed that the surface delaminations had appeared during grinding, but that was based on the assumption that sampling and metallographic preparation did not produce or propagate the defects. We no longer believe this is a safe assumption, so we will try to isolate these operations one at a time.

The platinum content in fabricated sheets prior to HD 427-432 has been higher than expected. When surface delaminations became a problem in the PICS, we decided to examine our starting powder more closely. The results shown in Table 4.2 indicated that only platinum and rhodium contents were significantly varying from lot to lot. Although there is no correlation of this data with the problem of surface delaminations in HD 427-432, the data indicate that our powder blending procedure needs improvement. One suggestion is that the as-received powders be homogenized in one large blender and then immediately separated into approximately 3-kg lots for storage. Any segregation of powders occurring during storage will then be eliminated when the dopants are blended into each lot just prior to compacting.

4.2.2 Foil

A total of 1900 cm² (290 in.²) of 0.12-mm (0.005-in.) Ir-0.3% W foil was shipped to MRC this quarter. Five hundred square centimeters (eighty square inches) of this foil was made primarily for development. In the past, the cold-rolled Ir-0.3% W foil shipped to MRC has been in the form of narrow strips, 20-50 mm (1-2 in.) wide, of random length. Mound Laboratory has requested that the foils be wider so that less foil is wasted in the blanking operation. Also, larger foils would help reduce our quality control costs.

When Ir-0.3% W is rolled with molybdenum covers to 0.25-mm (0.010-in.) foil, the centerline is normally thicker than the edges by about 0.02 mm (0.001 in.). This thickness variation causes splitting of the foil when the foil is subsequently cold rolled, usually within the first few bare rolling passes. Narrow pieces can be bare rolled to 0.12 mm (0.005 in.) without serious splitting or cracking because the thickness variation across the width will be less than that of wide pieces.

Wider foils can be obtained if the thickness of the foil is more uniform when it enters the bare rolling process. Samples of doped and undoped 0.25-mm (0.010-in.) foil were recrystallized and then bare rolled to 0.12 mm (0.005 in.). It was thought that if the bare rolling was started with the foil in a dead soft condition, then the stresses, caused by rolling material of nonuniform thickness, could not build up as high as in material

Table 4.2. Platinum and Rhodium Concentrations in HD-Series Powder and Sheet^a

Lots	Emission Spectroscopy		Spark Source Mass Spectrography		Fabricated Sheet ^b	
	Platinum	Rhodium	Platinum	Rhodium	Platinum	Rhodium
As-received, Platina-refined powder	—	—	50	60	—	—
HD 400-402 ^c	<50	<100	50	50	0.8	15
HD 403-408 ^c			500	50	25	30
HD 409-414 ^c	650	400	1500	300	25	30
HD 415-420 ^c			500	500	130	65
HD 421-426 ^c			500	250	110	40
HD 427-432 ^c	750	350	2000	250	60	25
HD 457-462 ^c	<100	<100	700	100	—	—
Remaining undoped powder			1000	700	—	—

^aConcentrations shown in ppm.

^bAverage concentration of lot.

^cPowder blended with dopants.

in the wrought condition. The undoped (WGR) foil delaminated badly, however, and failed the dye penetrant check. The doped (WGDR) foil rolled out well and was wider than most foils. However, MRC has reported a poor yield with this foil and metallography has revealed some apparent internal delaminations. Other foil samples were cross rolled in the bare rolling process. This allowed us to make wider foils, but their length was limited by the 250-mm- (10-in.-) wide rolling stand. The undoped (WGR) foil again delaminated badly and failed the dye penetrant check. The doped (WGDR) foil showed no delaminations either in dye penetrant or in metallography samples. MRC yield data is not available on this foil, but it appears that the cross-rolled foil does not offer any advantage over the narrow longitudinally-rolled foil in terms of yield. We have since found that by judiciously trimming the foil as we bare roll it in order to prevent the buildup of high stresses that split the foil, we can produce wider pieces, typically 60-mm (2 1/2-in.) wide. About eight foils will be obtained from each ingot by using this method instead of sixteen. Our yield per ingot will be about the same, and it is expected that MRC's yield per foil will improve.

4.3 Impact Properties of Production Material

C. T. Liu

We have characterized the impact properties of Ir-0.3% W production material with or without adding DOP-4 dopants. Sheet specimens were heat treated 19 hr at 1500°C and then impacted at 85 m/sec (280 fps) at 1350°C. Table 4.3 summarizes the impact data for doped (DOP-4) and undoped alloys. The preparation of these alloys is noted in the footnotes of this table. The undoped WG and WC had 9.6–10.7% elongation and 21–23% reduction of area at the impact conditions. All the doped material had better impact properties; however, their elongation varies from 15.2 to 26.8%, and their reduction of areas vary from 39 to 60%. Among the doped materials, DOP-4(0.3% W)-1 and HD-400 (prepared from 100% powder compacts) had the best impact properties, while WGD-271 and HD-416 and -428 (produced from 40% virgin compacts and 60% recycled skulls and heads through a successive electron-beam-melting process) had the worst impact properties. In regard to fracture behavior, WC and WG fractured mainly by grain-boundary separation.

Table 4.3. 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(0.3% W)-1 ^d	9.9	26.8	60	TF and DR
WGD-271 ^e	8.4	18.2	45	TF (Ma) and GBS (Mi)
HD-400 ^f	12.4	21.1	52	Mainly TF
HD-416 ^{g,h}	8.0	18.1	45	TF (Ma) and GBS (Mi)
HD-416 ^{g,h}		14.8	36	TF and GBS
HD-416 (av)		16.4 ± 2	40 ± 5	
HD-428 ^{g,h}	8.9	15.2	39	TF and GBS
HD-428 ^{g,h}		16.5	42	TF (Ma) and GBS (Mi)
HD-428 (av)		15.8 ± 2	40 ± 5	

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

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

^cUndoped Ir-0.3% W alloys.

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

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

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

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

^hDuplicate tests.

With an increase in ductility of the doped materials, the fracture mode changes from grain-boundary separation and transgranular fracture to transgranular fracture and ductile rupture. Table 4.3 also indicates that the production materials HD and WGD, produced through a successive melting process, had grain structure coarser than that of the materials produced from 100% powder compacts.

The results in Table 4.3 indicate that degradation of DOP-4 material occurs through the production process. This raises three critical questions: (1) What causes this degradation? (2) If degradation results from loss of dopants, which dopant element is responsible for this degradation? and (3) If the current production process continues, what are the expected (worst) impact properties of production material? The answers to these questions may be obtained from comparison of the data in Tables 4.3 and 2.1 (see p. 26 of this report). The impact properties and grain size of production material HD-428, -416, and WGD-271 are close to those of DOP-15 doped with thorium and iron* but with no aluminum. This suggests that the low-melting element, aluminum, is lost through evaporation during successive electron-beam melting, and that the poor impact properties of production material are most probably due to loss the the aluminum dopant. Furthermore, we believe that the lowest impact elongation of the doped production material should be close to that of DOP-15 (15% elongation after a heat treatment of 19 hr at 1500°C) and that this elongation is sufficient to survive the FSA impact test at maximum credible accident conditions.

Also note that sample HD-428 contained microscopic surface delaminations in the as-ground condition (see section 4.2 of this report), and they have no apparent effect on the tensile impact properties of that sample as compared to the other production lots having recycled feed material.

*The data indicate that doping with iron has no beneficial effect on impact properties and grain size of Ir-0.3% W alloy.

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J. R. Hadley
J. E. McCormick
- 48-49. Battelle Memorial Institute, 505 King Avenue, Columbus, OH 43201
C. Alexander
W. Pardue
50. Battelle's Pacific Northwest Laboratories, Battelle Blvd., Richland,
WA 99352
H. Fullam
51. Cabot Corporation, Stellite Division, 1020 Park Avenue, Kokomo, IN
46901
J. Tackett
52. Engelhard Industries, 700 Blair Road, Carteret, NJ 07008
H. J. Albert
53. Fairchild Space and Electronics Company, Germantown, MD 20767
A. Schock

54. General Electric Company, P.O. Box 15132, Cincinnati, OH 45215
R. G. Frank
55. General Electric Company, Valley Forge Space Center, P.O. Box 8048,
Philadelphia, PA 19101
E. W. Williams
56. Gulf Energy and Environmental Systems, P.O. Box 608, San Diego, CA
92112
N. Elsner
57. Kirtland Air Force Base, NM 87117
Directorate of Nuclear Safety
- 58-60. Los Alamos Scientific Laboratory, P.O. Box 1663, Los Alamos, NM
87545
R. D. Baker
S. E. Bronisz
S. Hecker
61. Minnesota Mining and Manufacturing Company, 2501 Hudson Road,
St. Paul, NM 55119
E. F. Hampl
- 62-63. Monsanto Research Corporation, P.O. Box 32, Miamisburg, OH 45342
W. T. Cave/D. P. Kelly
E. W. Johnson/D. L. Coffey
- 64-65. Savannah River Laboratory, E. L. DuPont de Nemours and Company,
Aiken, SC 29801
R. Gregg
J. R. Hilley
66. Sunstrand Energy Systems, 4747 Harrison Avenue, Rockford, IL 61101
E. Kreuger
- 67-68. Teledyne Energy Systems, 110 W. Timonium Road, Timonium, MD 21093
W. J. Barnett
W. Osmeyer
69. USERDA Dayton Area Office, P.O. Box 66, Miamisburg, OH 45342
E. A. Walker

- 70-82. USERDA Division of Nuclear Research and Applications, Washington, DC 20545
R. T. Carpenter	A. L. Mowery
T. J. Dobry	G. A. Newby
N. Goldenberg	B. J. Rock
T. J. Holleman	C. O. Tarr
W. D. Kenney	N. R. Thielke
A. P. Litman	E. J. Wahlquist
J. J. Lombardo	
83. USERDA Division of Reactor Research and Development, Washington, DC 20545
- J. M. Simmons
84. USERDA Oak Ridge Operations Office, P.O. Box E, Oak Ridge, TN 37830
- Research and Technical Support Division
85. USERDA Office of Program Coordination and Management — Reactors and Space, 1333 Broadway, Oakland, CA 94612
- W. L. Von Flue
86. USERDA Savannah River Operations Office, P.O. Box "A", Aiken, SC 29801
- L. M. East
- 87-113. USERDA Technical Informantion Center, Office of Information Services, P.O. Box 62, Oak Ridge, TN 37830
114. USNASA Headquarters, Washington, DC 20546
- T. B. Kerr
115. USNASA Jet Propulsion Laboratory, California Institute of Technology, 4800 Oak Grove Drive, Pasadena, CA 91103
- V. Truscello
- 116-117. USNASA Lewis Research Center, 2100 Brookpark Road, Cleveland, OH 44135
- M. Ault
- R. Migra
- R. H. Titran