OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION • NUCLEAR DIVISION
for the
U.S. ATOMIC ENERGY COMMISSION

ORNL - TM - 4424

Date: November 26, 1973

ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR DECEMBER 1971

R. G. Donnelly

NOTICE This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

CONTENTS

	<u>Page</u>
CLADDING MATERIALS PROGRAM	4
Preparation of Pt-Rh-W Sheet	4
Development of Improved Alloys	5
Characterization of Iridium	6
REFRACTORY ALLOYS PROGRAM	7
Effect of Oxygen Contamination on the Mechanical Properties of T-111	7
Effect of Oxygen on the Mechanical Properties of 0.090-in.-thick T-111	7
Effect of Oxygen Contamination on the Mechanical Properties of Molybdenum-Base Alloys	8
Effect of CO Contamination on the Mechanical Properties of Molybdenum-Base Alloys	9
MATERIALS COMPATIBILITY TESTING FOR THE LASL-DART PROJECT . . .	10

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

CLADDING MATERIALS PROGRAM

Preparation of Pt-Rh-W Sheet

J. H. Erwin

To compare the fabricability of the Pt-Rh-W alloy with small changes in composition, a drop-cast ingot approximately 1/8 in. thick by 1 in. wide by 2 in. long was made from each of three electron-beam-melted alloys with nominal compositions of Pt-26% Rh-8% W, Pt-30% Rh-10% W, and Pt-30% Rh-10% W-1% Hf-0.1% Ti. After the ingots were hot-rolled and then cold-rolled to 0.030-in.-thick sheet, standard 4T bend tests indicated low ductility in the Pt-2608 alloy sheet heat-treated at 950°C (stress relieved) and in both the Pt-3010 and the Pt-3010M modified alloys following heat treatment at 950 to 1200°C. However, the Pt-2608 alloy heat-treated 1000 and 1100°C (recrystallized) bent greater than 90° without visible cracking.¹

To better assure full recrystallization, an additional set of specimens were annealed at 100°C higher temperatures. Results of bend testing the alloy samples after the higher temperature heat treatment are given in Table 1. Heat treatment of the Pt-3010 and Pt-3010M alloys at 1200 and 1300°C did not appreciably improve the room-temperature elongation over that previously observed in samples heat-treated at 950 to 1100°C. We are presently metallographically examining these specimens in an attempt to determine the reason for these low-bend ductilities.

Table 1. Bend Formability Evaluation of Recrystallized Pt-Rh-W Alloys at Room Temperature

	Alloy		
	Pt-2608	Pt-3010	Pt-3010M
Annealing temperature, °C	1200	1200	1300
Minimum R/t for 90° bend (calc) ^a	2.7	9.2	9.3
Bend elongation (% calc) ^a	15.5	5.2	5.1
Bend strength, psi (calc) ^a	144,000	117,000	206,000

^aCalculations according to equations set forth by W. W. Wood *et al.* in Theoretical Formability, ASD TR 61-191, vol. II.

In an effort to determine the maximum bending permissible in multi-stage forming operations, two samples of the Pt-2608 alloy were annealed at 1200°C, subjected to a 45° 1T bend, reannealed, and the bend continued to

¹R. G. Donnelly, *Isotopic Power Materials Development Report for November 1971*, ORNL CF-71-12-20, Oak Ridge National Laboratory.

100°. Data from previous samples indicated an allowable 1T bend angle of 42.6° for this alloy. Microscopic inspection at 20X following the initial and final bends revealed no cracking; however, examination of the maximum bend area with the aid of the scanning electron microscope at 1000X revealed indications of grain boundary separation at the surface. As a result we feel that no problem should be encountered in the room-temperature fabrication of 1200°C annealed Pt-2608 alloy with per pass elongation typically less than 15%.

Development of Improved Alloys

C. T. Liu

The tensile properties of hafnium- and titanium-stabilized Pt-Rh-W alloys were determined at room temperature and in vacuum at 760 and 1093°C. The results are presented in Table 2 with previously reported data.² The hafnium and titanium additions improve the tensile strength of Pt-Rh-W base at all temperatures measured. This effect is most prominent at 1093°C. The room-temperature ductility of stabilized alloys is lower than that of the base; however, 1% Hf and 0.2% Ti improve the ductility of Pt-30% Rh-8% W alloy from 18 to 24.1% at 1093°C.

Table 2. Room and Elevated-Temperature Tensile Properties of the Platinum-Base Alloys Recrystallized 1 hr at 1200°C

Alloy Composition (wt %)	Ultimate Tensile Strength (psi)	Elongation (%)
<u>Room Temperature</u>		
Pt-30 Rh	-	-
Pt-30 Rh-6 W	112,000	26
Pt-26 Rh-8 W	-	-
Pt-30 Rh-10 W	118,000	14.5
Pt-30 Rh-6 W-0.5 Hf-0.2 Ti	113,000	19.3
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	135,000	19.0
Pt-30 Rh-8 W-1 Hf-0.2 Ti	130,000	11.7
<u>760°C (1400°F)</u>		
Pt-30 Rh	48,000	28.8
Pt-30 Rh-6 W	80,000	23.3
Pt-26 Rh-8 W	-	-
Pt-30 Rh-10 W	95,000	28

²R. G. Donnelly, *Isotopic Power Materials Development Report for October 1971*, ORNL CF-71-11-27, Oak Ridge National Laboratory

Table 2. continued

Alloy Composition (wt %)	Ultimate Tensile Strength (psi)	Elongation (%)
Pt-30 Rh-6 W-0.5 Hf-0.2 Ti	81,000	27.5
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	91,000	24.3
Pt-30 Rh-8 W-1 Hf-0.2 Ti	108,000	26.3
<u>1093°C (2000°F)</u>		
Pt-30 Rh	24,000	38
Pt-30 Rh-6 W	36,000	33.3
Pt-26 Rh-8 W	38,000	18
Pt-30 Rh-10 W	47,000	15.5
Pt-30 Rh-6 W-0.5 Hf-0.2 Ti	42,200	28.1
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	44,500	12.5
Pt-30 Rh-8 W-1 Hf-0.2 Ti	52,000	24.5

Characterization of Iridium

C. T. Liu

The recrystallized iridium sheet specimens are continuously oxidized in static air at 1040, 900, and 770°C. At present, the oxidation runs have logged 1100 hr. The results are described below.

Oxidation at 1040°C - No oxide layer is observed at this temperature. After 50 hr exposure, the material on the edge and surface of the specimen gradually loses its adhesion and begins to flake in the form of metallic grains. However, the oxidation rate is quite linear, at an average rate of -880×10^{-6} g/cm²·hr. This value is in excellent agreement with the reported value (-900×10^{-6} g/cm²·hr) by Douglass *et al.*³

Oxidation at 900°C - An adherent dark blue oxide layer gradually appears on the specimen surface at 900°C. The initial stage of oxidation is parabolic with a low oxidation rate. However, after 600 hr exposure, the specimen begins to show corner effects and the oxidation rate increases to approximately -27×10^{-6} g/cm²·hr.

Oxidation at 770°C - Although the dark blue oxide layer is observed to form, no apparent weight change can be measured at this temperature.

³R. W. Douglass *et al.*, *High Temperature Properties and Alloying Behavior of the Refractory Platinum-Group Metals*, NP-10939, Battelle Memorial Institute (August 1961).

REFRACTORY ALLOYS PROGRAM

Effect of Oxygen Contamination on the Mechanical Properties of T-111

C. T. Liu

The effects of specimen thickness and doping temperature on the mechanical properties of oxygen-contaminated T-111 were reported previously.⁴ We know that the room-temperature ductility of 40- and 20-mil specimens is reduced to about 1.5% when contaminated with 300 and 650 ppm oxygen, respectively. In order to determine the effect of testing temperature, the contaminated mechanical property specimens were tested at temperatures up to 1093°C; the results are presented in Table 3. The specimens have higher ductility at 825°C and lower ductility at room temperature and 1093°C. The results clearly indicate that there is no "ductile-to-brittle transition temperature" existing in the oxygen-contaminated T-111; instead, there is a "ductility maximum" that fortuitously appears to occur at approximately 825°C.

Table 3. Effect of Testing Temperature on the Tensile Properties of 40- and 20-mil Sheet T-111 Specimens Doped with Oxygen at 825°C

Specimen Thickness (mil)	Oxygen Amount (ppm)	Testing Temperature (°C)	Elongation (%)	Tensile Strength (psi)
40	300	Room Temperature	1.6	96,000
	296	825	10.2	66,000
	306	1093	2.5	51,000
	330	1093	2.4	51,000
20	641	Room Temperature	1.3	125,000
	660	825	2.0	70,000
	680	1093	0.5	77,000

Effect of Oxygen on the Mechanical Properties of 0.090-in.-thick T-111

H. Inouye

The study on the effect of oxygen contamination on the tensile properties of 90-mil T-111 at low doping rates has continued. As previously reported, the ductility of T-111 at 825°C is insensitive to the contamination level up to a critical level. Above the critical level, the ductility decreases sharply with the oxygen content.⁵

⁴R. G. Donnelly, ORNL CF-71-12-20.

⁵R. G. Donnelly, ORNL CF-71-12-20.

Table 4 lists the tests to date on oxygen-contaminated T-111. Up to 290 ppm oxygen, the tensile strength increased with the oxygen content but did not impair the ductility at all. These results show that the critical oxygen level has not been reached at the 290-ppm level. The higher than expected ductility based on 20- and 40-mil-thick specimens may be due to the lower doping rate and/or the elimination of oxygen from the edges of these tensile specimens by machining.

Table 4. Effect of Oxygen on Tensile Properties of 90-mil-thick T-111 at 825°C

Amount of Oxygen Added (ppm)	Ultimate Tensile Strength (psi)	Elongation (%)
Control ^a	59,200	18.0
185	72,500	20.5
290	77,500	18.5

^aAnnealed 1 hr at 1650°C. Annealed analysis:
10 ppm oxygen, 10 ppm nitrogen, 3 ppm hydrogen, and
24 ppm carbon.

Effect of Oxygen Contamination on the Mechanical Properties of Molybdenum-Base Alloys

C. T. Liu

In order to evaluate the use of molybdenum-base alloys as potential cladding materials for space isotopic heat sources, TZM sheet specimens were doped with oxygen at 825°C and 1×10^{-5} torr oxygen pressure. At present, the contamination has accumulated to 2000 hr; the tensile data at room and elevated temperatures are presented in Table 5. The oxygen doping of TZM at 825°C only causes a small increase of tensile strength and a moderate decrease of ductility. As a matter of fact, TZM exhibited 24.5% strain at room temperature and 17.7% at 825°C after 2000 hr exposure. Therefore, we conclude that TZM is compatible with low pressure oxygen under these conditions.

Table 5. Tensile Properties of 20-mil-thick TZM Sheet Specimens Contaminated with Oxygen at 825°C

Doping Conditions		Elongation	Ultimate Tensile
Time (hr)	Oxygen Pressure (torr)	(%)	Strength (psi)
<u>Room Temperature</u>			
As recrystallized		37	79,000
As recrystallized		33.5	79,600
100	4×10^{-5}	35.3	81,200
500	1×10^{-5}	29.2	85,000
2,000	1×10^{-5}	24.5	85,000
<u>825°C</u>			
As recrystallized		24.8	45,000
2,000	1×10^{-5}	17.7	51,200

Effect of CO Contamination on the Mechanical Properties of Molybdenum-Base Alloys

C. T. Liu

The 20-mil sheet specimens of TZM were contaminated at a CO pressure of 1×10^{-5} torr and 825°C to 2000 hr. The tensile data at room and elevated temperatures are summarized in Table 6. Both the tensile strength and elongation of the contaminated specimens are not significantly different from the as-recrystallized ones, even after 2000 hr exposure. We therefore conclude that TZM is compatible with CO gas under these conditions. These results together with the results of oxygen contamination reported in the previous section lead to the general conclusion that, from the environmental stability standpoint, TZM would be a suitable cladding material for SNAP-19 radioisotope thermoelectric generators operating at 800 to 850°C.

Table 6. Tensile Properties of 20-mil-thick TZM Sheet Specimens Contaminated with CO Gas at 825°C and 1×10^{-5} torr Oxygen Pressure

Doping Time (hr)	Elongation (%)	Ultimate Tensile Strength (psi)
<u>Room Temperature</u>		
As recrystallized	37.0	79,000
As recrystallized	33.5	79,600
196	33.2	79,500
1,000	30.0	83,000
2,000	27.9	82,000

Table 6. continued

Doping Time (hr)	Elongation (%)	Ultimate Tensile Strength (psi)
<u>825°C</u>		
As recrystallized	24.8	45,000
2,000	22.5	46,300

MATERIALS COMPATIBILITY TESTING FOR THE LASL-DART PROJECT

J. R. DiStefano and A. C. Schaffhauser

Samples of Ta, W, Mo, Re, Mo-50% Re, Pt-Rh-W, and BeO were exposed to graphite for 500 hr at 1300, 1400, and 1500°C. Visual observations were reported previously.⁶ Samples tested at 1400°C have been submitted for metallographic evaluation and will be examined shortly.

The metal-oxide and metal-metal compatibility tests scheduled for 500 hr at 1300, 1400, and 1500°C have been completed.⁶ After visual examination, samples tested at 1400°C will be submitted for chemical and metallographic analysis.

⁶R. G. Donnelly, ORNL CF-71-11-27.