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ISOTOPIC POWER MATERIALS DEVELOPMENT PROGRESS REPORT FOR NOVEMBER 1971

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CLADDING MATERIALS PROGRAM

Preparation of Pt-3010 Sheet

J. H. Erwin

To compare the fabricability of the Pt-3010 alloy with small changes in composition, 500-g ingots of Pt-26% Rh-8% W, Pt-30% Rh-10% W, and Pt-30% Rh-10% W-1% Hf-0.1% Ti were electron-beam melted and cast into pancakes. These castings cracked while being hot worked (1075-1100°C) and were remelted in the electron-beam furnace and drop cast.¹

All three of the drop-cast ingots were annealed 48 hr at 1050°C in an air atmosphere followed by 92% hot reduction to a thickness of 0.040 in. The Pt-2608 alloy was hot-rolled at 1200°C. The other two alloys were hot-rolled at 1250-1300°C. Each hot-rolled plate was sheared to approximately one-half length. One piece of each was stored for future processing and the other annealed 15 min in air at 1200°C followed by cold-rolling to 0.030-in. thickness. The cold-rolling direction was at 90° with respect to the hot-rolling direction. Intermediate anneals were not necessary for cold-rolling the Pt-2608 alloy, but were after 10-15% reduction for the Pt-3010 alloy and after 5-10% reduction for the Pt-3010 modified alloy.

Following cold rolling, both transverse and longitudinal (as related to cold rolling) samples were taken from each alloy. These samples were heat treated for 1 hr at 950-1200°C in vacuum to obtain varied grain structures for bend testing. After heat treatment, the surface to be put in tension during bending and the two edges were polished to remove all visible imperfections. The data from 4T bend tests conducted at room temperature and 0.2 in./min are summarized in Table 1.

The Pt-2608 alloy heat treated to 1000 and 1100°C bent >90° without visible cracking. The Pt-3010 and modified Pt-3010 specimens all broke at bend angles of 13 to 37°. The specimens of both alloys heat treated at the higher temperatures exhibited improved ductility over the other samples. For this reason we are investigating a 100°C higher annealing temperature for these two alloys.

¹R. G. Donnelly, *Isotopic Power Materials Development Progress Report for October 1971*, ORNL CF-71-11-27, Oak Ridge National Laboratory.

Table 1. Summary of 4T Room-Temperature Bend Data for Pt-Rh-W Alloys

Sample Number	Heat Treatment (°C)	Bend Angle ^a	Outer Fiber Bend Strength (psi)
<u>Pt-2608</u>			
1-1L	950	35	256,000
1-1T	950	33	261,000
1-3L	1000	>90	245,000
1-3T	1000	>90	231,000
1-5L	1100	>90	161,000
1-5T	1100	>90	152,000
<u>Pt-3010</u>			
2-1L	950	26	210,000
2-1T	950	19	211,000
2-3L	1000	21	219,000
2-3T	1000	18	212,000
2-5L	1100	37	203,000
2-5T	1100	24	198,000
<u>Pt-3010M</u>			
3-1L	950	15	360,000
3-1T	950	14	343,000
3-3L	1050	15	346,000
3-3T	1050	13	342,000
3-5L	1200	21	216,000
3-5T	1200	21	236,000

^aTotal angle that specimen was bent or angle at specimen failure.

Development of Improved Alloys

C. T. Liu

In order to show the effects of hafnium and titanium on the physical and mechanical properties of the Pt-Rh-W alloy base, four ingots with compositions Pt-30% Rh-8% W-1% Hf-0.2% Ti, Pt-30% Rh-8% W-0.5% Hf-0.2% Ti, Pt-30% Rh-8% W-0.25% Hf-0.1% Ti, and Pt-30% Rh-6% W-0.5% Hf-0.2% Ti were prepared by either electron-beam drop-casting or casting into pancake form. The ingots were first hot-rolled in air in the 1000 to 1230°C range. When the as-cast structure was broken, the alloys were successfully rolled at room temperature to 20- to 35-mil sheet with intermediate anneals between 950 and 1000°C.

Tensile specimens were stamped from the sheets and tested at room and elevated temperatures. Table 2 shows the tensile properties of Pt-30% Rh-8% W-1% Hf-0.2% Ti and Pt-30% Rh-8% W-0.5% Hf-0.2% Ti alloys in the

recrystallized condition. The data for Pt-Rh-W alloys reported previously² are included for comparison. Some significant points to be noted are: (1) The Hf- and Ti-stabilized alloys are much stronger than the base ternary alloy, even up to 1093°C. The 0.5% Hf-stabilized alloy is comparable to, and the 1% Hf alloy is stronger than Pt-30% Rh-10% W. (2) Table 2 indicates that 0.5% Hf improves and 1% Hf impairs the room-temperature ductility of the alloy, but at elevated temperature the ductility is not sensitive to the hafnium content. (3) Alloying with hafnium and titanium improves the ductility of base Pt-Rh-W alloy 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	a	a
Pt-30 Rh-6 W	112,000	26
Pt-26 Rh-8 W	a	a
Pt-30 Rh-10 W	118,000	14.5
Pt-30 Rh-8 W-1 Hf-0.2 Ti	130,000	11.7
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	135,000	19.0
<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	a	a
Pt-30 Rh-10 W	95,000	28
Pt-30 Rh-8 W-1 Hf-0.2 Ti	108,000	26.3
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	91,000	24.3
<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-8 W-1 Hf-0.2 Ti	52,000	24.1
Pt-30 Rh-8 W-0.5 Hf-0.2 Ti	a	a

^aNot tested.

²R. G. Donnelly, *Isotopic Power Program Materials Development Progress Report for August 1971*, ORNL CF-71-9-5, Oak Ridge National Laboratory.

Characterization of Iridium

C. T. Liu

Last month's progress report³ indicated that warm-rolled iridium sheet had the best bend ductility after annealing 1 hr at 1200°C. Accordingly, the iridium sheet was heat treated at that temperature, and tensile specimens were stamped out successfully at room temperature. Tensile test results at room and elevated temperatures are shown in Table 3 for specimens recrystallized 1 hr at 1500°C. In this condition, iridium has quite low tensile strength at room temperature, and its strength decreases slowly with temperature. The strength data in Table 3 are in good agreement with the results for commercially pure iridium reported by Jaffee *et al.*⁴ It should be noted that iridium is much weaker than the hafnium- and titanium-stabilized Pt-Rh-W alloys as shown in Table 2.

The ductility of recrystallized iridium is low at room temperature but increases to 23.5% at 760°C. This result is also consistent with Jaffee's work where he found the ductile-to-brittle transition temperature of iridium to be approximately 600°C.

Table 3. Room- and Elevated-Temperature Tensile Properties of the Pure Iridium Metal Recrystallized 1 Hr at 1500°C

Testing Temperature (°C)	Ultimate Tensile Strength (ksi)	Elongation (%)
Room Temperature	56	5.7
760	56	23.5
1093	35	19.8

REFRACTORY ALLOYS PROGRAM

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

C. T. Liu

In order to show the effects of specimen thickness and doping temperature on the mechanical properties of oxygen-contaminated T-111, both 20- and 40-mil sheet specimens were doped with oxygen at 825°C (the operation

³R. G. Donnelly, ORNL CF-71-11-27.

⁴R. I. Jaffee *et al.*, "Rhenium and the Refractory Pt-Group Metals," p. 383 in *Refractory Metals and Alloys*, vol. II, ed. by M. Semchyshen and J. J. Harwood, Interscience, New York, 1961.

temperature of Pioneer F) and 1×10^{-5} torr oxygen pressure. The tensile properties at room temperature and 825°C are summarized in Tables 4 and 5. The ductilities obtained at room and elevated temperatures are also plotted, respectively, in Figs. 1 and 2 as a function of amount of oxygen added by doping at 825 and 1000°C. As indicated in the tables, the tensile strength increases with increasing oxygen. Figure 1 shows that the room-temperature ductility of T-111 doped at 1000°C decreases continuously with oxygen up to 700 ppm, and that doping at 825°C causes only a moderate decrease of ductility at low oxygen levels and sharp decrease at higher levels. The 20-mil specimens can tolerate about 100 ppm more oxygen than 40-mil specimens. When tested at elevated temperatures (Fig. 2), the ductility keeps constant initially until a critical oxygen level is reached. The critical oxygen level varies with both the doping temperature and specimen thickness. Beyond that level, the ductility decreases continuously with oxygen content. For a strain of 5%, the T-111 doped at 825°C can tolerate 480 and 560 ppm for 40- and 20-mil specimens, respectively.

Fig. 1. Room-Temperature Ductility of T-111 Specimens As a Function of the Amount of Oxygen Doped at 825 and 1000°C.

The results so far indicate that both doping temperature and specimen thickness have a big effect on the properties of oxygen-contaminated T-111. It is also expected that the mechanical behavior of T-111 varies with the doping rate, because the doping rate affects the surface oxygen concentration and the oxygen gradient. It should be noted that the above data were obtained from specimens doped at a high rate (40 to 100 ppm/hr), and that different results may be obtained if different rates are used.

Fig. 2. Elevated-Temperature Ductility of T-111 Specimens As a Function of the Amount of Oxygen Doped at 825 and 1000°C.

Table 4. Tensile Properties of 20-mil Sheet T-111 Specimens As-Doped with Various Levels of Oxygen at 825°C

Oxygen Amount Doped (ppm)	Elongation (%)	Ultimate Tensile Strength (psi)
<u>Room Temperature</u>		
0	28	101,000
302	19	111,000
400	2	100,000
641	1.3	125,000
770	0.8	150,000
<u>825°C</u>		
0	17.8	69,000
290	15	79,000
375	10.6	81,000
460	8.5	83,000
565	4.0	71,000
660	2.0	70,000

Table 5. Tensile Properties of 40-mil Sheet T-111
Specimens As-Doped with Various Levels of Oxygen at 825°C

Oxygen Amount Doped (ppm)	Elongation (%)	Ultimate Tensile Strength (psi)
<u>Room Temperature</u>		
162	25.5	99,500
215	20.5	106,000
300	1.6	96,000
391	0.5	114,000
<u>825°C</u>		
140	18	64,000
296	10.2	66,000
389	7.5	63,000
510	4.2	69,000

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

H. Inouye

Previously, we reported that the oxygen concentration causing embrittlement of T-111 decreased as the doping temperature was decreased from 1000 to 825°C (ref. 5) and also as the specimen thickness was increased from 20 mils to 40 mils.⁶ The doping rate of the above specimens at 825°C were also higher (80 ppm/hr for 20-mil specimens and 40 ppm/hr for the 40-mil specimens) than that indicated in the Pioneer heat source. Because of these differences, a new series of 90-mil-thick specimens are being doped with oxygen at a rate of 1.5 ppm/hr (1×10^{-6} torr oxygen at 825°C). In addition to these changes, the oxygen is being added to blanks 0.090 x 3/4 x 2-1/2 in. prior to machining the tensile specimens in order to eliminate oxygen from the edges of the specimen gage length which was present in the thinner specimens.

The tensile properties of 90-mil specimens at 825°C tested to date are presented in Table 6.

⁵E. Lamb, *IPF Monthly Status Report for September 1971*, ORNL CF-71-10-34, Oak Ridge National Laboratory.

⁶See Figs. 1 and 2 pp. 8 and 9.

Table 6. Tensile Properties of 90-Mil Specimens Tested at 825°C

Condition	Ultimate Tensile Strength (psi)	Elongation (%)
As received ^a	59,200	18.0
Doped with 185 ppm oxygen	72,500	20.5

^aOxygen content 10 ppm, annealed 1 hr at 1650°C.

The above results only show an increase in the ultimate tensile strength and no deleterious effect on ductility. These results are similar to those obtained for 20- and 40-mil specimens and indicate that the doping rate and specimen thickness are not variables influencing the ductility at this oxygen level.

Contamination of T-111 and TZM by Impurities Outgassed from Graphite and Min-K 1301

H. Inouye

Tensile specimens of T-111, molybdenum-coated T-111, and TZM continue to be exposed to the simulated Pioneer environment described previously.⁷ The room-temperature tensile tests conducted to date are summarized in Table 7. Bare T-111 shows complete embrittlement as would be expected for specimens containing several thousand parts per million oxygen. The edges of the T-111 specimen coated with 0.0002 in. of evaporated molybdenum fragmented into a black powder and consumed about one-half of the tensile specimen after 1030 hr of exposure. This specimen was unsuitable for tensile testing. The T-111 specimen coated with 0.0002 in. of evaporated molybdenum plus 0.001 in. of CVD molybdenum failed after 802 hr of exposure at coating defects in the specimen shoulders in the manner described above. The intact gage section of this specimen was bent without failure through a 1T bend at room temperature. From this result, the ductility in the outer fiber of the 20-mil specimen was estimated to be approximately 30% and the oxygen contamination was estimated to be <100 ppm.

The results for TZM clearly show that the simulated Pioneer environment has no effect for exposures to 1030 hr at 825°C. Scheduled exposures to 2000 hr for additional TZM specimens have logged approximately 1500 hr (as of November 29, 1971).

⁷M. Semchyshen and R. Q. Barr, *Symposium on Newer Metals*, Special Technical Publication No. 272, ASTM (1959), pp. 1-24.

Table 7. The Effect of Impurities Degassed from Graphite and Min-K 1301 on the Room Temperature Tensile Properties^a of 20-mil-thick T-111 and TZM

Material	Exposure (hr)	Oxygen Contamination (calculated ppm)	Ultimate Tensile Strength (psi)	Elongation (%)
T-111	204	1900	113,000	0 ^b
T-111	534	6480	Fractured on handling	
0.1 mil Mo on T-111	204	820	139,000	0 ^b
0.2 mil Mo on T-111	1030	—	Fragmented in environment	
1.2 mil Mo on T-111	802	<100 (est)	—	~30 (est) ^c
TZM	534	Not determined	77,800	35.7
TZM	1030	Not determined	78,400	32.8
TZM	Control	—	79,600	33.5

^aSpecimen at 825°C; graphite and Min-K at 538°C; environment of 600 torr argon plus impurities.

^bFractured in shoulder of specimen

^cBased on bend test.

Contamination Studies of TZM by Water Vapor

H. Inouye

The effect of water vapor in the tensile properties of TZM is being evaluated since this gas is a probable impurity in the Pioneer generators. Exposures of 20-mil TZM specimens (approximate weight = 2.4 g) to 1×10^{-5} torr water vapor at 825°C show an average weight loss of 0.0005 g after 457 hr. At 1002 hr of exposure, a very small weight increase of 0.000032 g was measured. Evidences of any reaction was not visible.

Table 8 shows the tensile properties of TZM after exposure to water vapor. The room-temperature tests show a small decrease in the elongation that appears to become more serious with increasing exposure time. The tensile strengths of the exposed specimens are also higher than the control. Although the tests at 825°C show evidence of some loss of ductility, elongation of ~16% is characteristic of recrystallized and uncontaminated TZM at 982°C.⁷ Scheduled exposures of TZM to 2000 hr have logged approximately 1500 hr (as of November 29, 1971).

Table 8. Tensile Properties of 20-mil-thick TZM Exposed to 1×10^{-5} Torr Water Vapor at 825°C

Test Temperature (°C)	Exposure (hr)	Ultimate Tensile Strength (psi)	Elongation (%)
Room	Control	79,600	33.5
Room	457	83,200	31.0
Room	1002	80,600	29.3
825	Control	45,000	24.8
825	457	45,600	20.2
825	1002	46,000	16.3

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

C. T. Liu

In order to qualify the use of a Mo-Re alloy as the cladding material for space isotopic heat sources, Mo-46% Re sheet specimens were doped with oxygen at 825°C and 1×10^{-5} torr oxygen pressure. The contaminated specimens were then tensile tested at room temperature and 825°C; the results are listed in Table 9. Although the specimens showed a weight gain corresponding to approximately 800 ppm after the 1000-hr exposure, Table 9 indicates no change in the mechanical properties due to the oxygen contamination.

Table 9. Tensile Properties of 20-mil-thick Mo-46% Re Sheet Specimens Doped with Oxygen at 825°C and 1×10^{-5} Torr Oxygen Pressure

Doping Time (hr)	Testing Temperature (°C)	Elongation (%)	Ultimate Tensile Strength (psi)
As recrystallized	Room	8.2	195,000
1000	Room	9.2	192,000
1000	825	13.7	116,000

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

C. T. Liu

The 20-mil sheet specimens of TZM and Mo-46% Re alloys were contaminated at a CO pressure of 1×10^{-5} torr and at 825°C. The contaminated specimens were then tensile tested at room temperature. The results shown in Table 10 indicate that the tensile properties of the contaminated specimens are not significantly different from the as-recrystallized ones. Based on the limited data, we concluded that the molybdenum-base alloys are compatible with this concentration of CO gas at 825°C for at least 1000 hr.

Table 10. Effect of CO-Gas Contamination on the Room-Temperature Tensile Properties of TZM and Mo-46% Re

Alloy	Doping Time (hr)	Elongation (%)	Ultimate Tensile Strength (psi)
TZM	a	33.5	79,000
TZM	196	33.2	79,500
TZM	1000	30.0	83,000
Mo-46% Re	a	8.2	195,000
Mo-46% Re	1004	10.7	190,000

^a As recrystallized.

Examination of Pioneer Heat Source Capsule PF-3

E. L. Long, Jr.

The results of the examination of the T-111 specimens from Pioneer heat source capsule PF-7 and an archive specimen were reported last month. Since that time bend specimens prepared from capsule PF-3, as well as PF-7 and the archive capsule, have been tested. We selected one of the bend specimens (viz., No. 5) from PF-3 for a fractographic examination with the aid of a scanning electron microscope. Examination of the fracture surface disclosed that the fracture mode was a transgranular cleavage with no evidence of ductile fracture. There was no variation in the appearance of the fracture surface across the wall of the capsule.

Pioneer Capsule Bend Test Results

B. McNabb and H. E. McCoy

Bend tests were conducted on specimens from an archive sample and two specimens each from PF-3 and PF-7. The specimens were about 1/8 in. wide by 0.090 in. thick by 3/4 in. long. They were cut by abrasive cutoff wheel, without coolant, and some specimens discolored slightly, particularly archive samples 1 and 2. This discoloration in samples 1 and 2 make the data from them questionable.

The bend tests were run on an Instron Tensile Machine using a bend test fixture 1/2 in. between end supports, and a 3/16-in.-diam rod as the nose piece of the ram. The tests were run at room temperature with a crosshead speed of 0.05 in./min, chart speed of 5 in./min, and load range of 0-500 lb.

The specimens were cut from about 2-in.-ID by 0.090-in. piece with the length of the specimen aligned with the length of the original capsule. The specimens were treated as flat sheet in calculating the stress and strain and the slight curvature in the 1/8 in. width was ignored. One specimen each was tested with the outside diameter and inside diameter of the capsule in tension from each test capsule and the archive capsule.

Table 11 shows the bend data. Several stress parameters are reported for comparison purposes. The proportional limit is the nearest to the true value, but the other stresses become progressively in error since elastic formulas were used for computing the stresses when the sample was actually deforming plastically. The stress values reported are outer fiber stresses and are approximately a factor of 2 greater than equivalent bulk tensile properties.

The specimens from PF-3 exhibited no measurable plastic strain or deviation from the elastic portion of the stress-strain curve. However, as shown in the table, they did sustain a substantial load. The specimens from PF-7 were much more ductile. The specimen tested with the inside diameter of the tube in tension did not fail, but was discontinued

after 0.4 in. crosshead travel (90° bend angle). The specimen with the outside diameter in tension did fail after a deflection of 0.24 in. (52° bend angle). The archive specimens showed good ductility with the specimens that were not discolored during cutting being more ductile.

Table 11. Results of Room-Temperature Bend Tests on T-111 from Pioneer Capsules PF-3, PF-7 and an Archive Capsule

Sample Number	Side in Tension	Stress (ksi)			Fracture	Crosshead Travel (in.)	Bend Angle
		Proportional	Yield ^a	Maximum			
PF3 - 5	ID	—	—	94.4	yes	0.013	3
PF3 - 6	OD	—	—	132	yes	0.019	4
PF7 - 8	OD	137	154	161	yes	0.243	52
PF7 - 9	ID	144	151	211	no	0.401	90
Archive-3	ID	152	158	203	no	0.50	90
Archive-4	OD	115	140	213	no	0.40	90
Archive-1 ^b	ID	109	144	251	yes	0.178	40
Archive-2 ^b	OD	98.2	132	235	yes	0.135	30

^aBased on deflection offset of 0.002 in.

^bSpecimens were discolored during abrasive cutting.