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PLUTONIA/CURIA COMPATIBILITY TESTING

OCTOBER - DECEMBER 1972

QUARTERLY REPORT NO. 17

J. D. WATROUS

PREPARED FOR THE ATOMIC ENERGY COMMISSION,
DIVISION OF SPACE NUCLEAR SYSTEMS,
UNDER CONTRACT NO. AT(45-1)-2138

DATE PUBLISHED - MARCH 1973

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Donald W. Douglas Laboratories
Richland, Washington

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SUMMARY

Electron-beam microprobe analyses reconfirmed vapor transport of both rhenium and plutonia in Task 1 test capsules exposed at 1800°C for 15,000 hr or more. Effects are to reduce temperature gradients within the capsule and also to remove metal from hot areas. The microprobe has also been used to determine that titanium and hafnium transport readily between Task 2 refractory metal alloy containers at 1800°C, forming massive precipitates. This transport did not occur after 5000 hr at 1500°C, and in all probability current or proposed space thermoelectric systems would not be affected at typical operating temperatures of 900° to 1200°C. Limited probe work remains on Tasks 1 to 3 materials to complete the current effort on these tasks.

Visual and metallographic examination has been completed on Task 5 Multi-Hundred Watt (MHW) mockup test assemblies; MHW is being developed for the LES 8/9 mission. Plugging of the vent through the iridium container occurred in two of three capsules exposed at maximum temperatures of 1440°C for 1250 hr. Plugging was apparently caused by iridium transport into the 0.0008-in. diameter electron-beam-drilled hole. Black deposits, believed to be plutonia also transported, especially above the fuel but not in massive amounts or discrete layers. Graphite erosion occurred immediately opposite the vent hole, but no other degradation was observed in the graphites. A thin reaction layer was observed in the iridium in contact with the plutonia as a result of tungsten present in the plutonia, which did not appear to result in damage to the bulk mechanical properties of the iridium.

Microsize iridium tensile specimens, exposed in contact with either plutonia or graphite, retained both strength and ductility equivalent to comparably exposed control specimens when tested at a high strain rate and expected impact temperature of 1400°C. Strength remained the same as control specimens which were not exposed to plutonia. Ductility was reduced from 58% elongation for control specimens to 49% elongation for specimens exposed at ~1370°C with and without a subsequent thermal pulse to 1950°C. A number of tensile specimens exposed at somewhat lower temperature (~1200°C) exhibited reduced ductility in the lower temperature (~1000°C) areas of the specimen.

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Section 1

INTRODUCTION

Plutonia/curia compatibility testing at the Donald W. Douglas Laboratories (DWDL) has evolved from simple oxide/monometallic configurations to complex mockups of engineering systems. Emphasis has changed from determining basic reaction mechanisms to ensuring that future space radioisotope thermoelectric systems will function as intended. Essentially all effort has been completed on earlier work with current experimental effort almost totally devoted to the Multi-Hundred Watt (MHW) mockup test program. MHW is being developed by SNS for the LES 8/9 mission.

Since October 1968, this program has expanded into five tasks. Task 1 effort studied reactions between small plutonia-238 and curia-244 pellets in single refractory alloy containers at 1800° and 2000°C and determined stability of a plutonia-molybdenum cermet precursor of the current plutonium-molybdenum cermet (PMC) fuel. Task 2 was structured to determine stability of the later PMC used in the Pioneer and Transit missions, and solid-solution cermet (SSC) fuel forms and to evaluate interrelationships with double refractory alloy containment systems at 1500°, 1800°, and 2000°C. Task 3 was designed to give early information on potential MHW materials combinations with potentially useful barriers interposed between the plutonia fuel and iridium or platinum alloy containers exposed at 1500°C. Task 4 was added to study aging characteristics of plutonia and PMC fuel forms using curium-242 additions to accelerate helium generation. Experimental work has been completed on Tasks 1 through 4, except for a limited number of electron-beam microprobe analyses to be completed. Topical reports will then be prepared concluding these tasks.

Task 5 was added to study MHW heat source systems interrelationships mocked-up in a subsize configuration, including iridium tensile specimens for determining mechanical behavior after thermal treatment. Four plutonia-fueled and three control (non-fueled) thermal-gradient test specimens were assembled. Each contained microsize (0.75 in length) iridium tensile specimens, some adjacent

to the fuel, others in contact with either the graphic impact liner or graphite ablator materials. Two of each of these test have been evaluated during the current quarter. Three plutonia powder fueled and three control uniform-temperature test capsules containing subsize (1.5 in length) iridium tensile specimens were also assembled with one of each partially evaluated during the current quarter. Considerable effort was expended on this task to provide information to the MHW program for a comprehensive design review meeting scheduled for early 1973.

Section 2

RESULTS

Electron-beam microprobe analyses were completed on 4 of 14 specimens from Tasks 1, 2, and 3. A radioactive spill at Westinghouse Hanford Company, subsequent cleanup, and more-stringent handling procedure approval delayed further microprobe work during the entire quarter. Visual and metallographic evaluation of three plutonia-fueled and three control Task-5 MHW mockup test assemblies were almost completed during the quarter. High-strain-rate tensile tests were completed on the accompanying series of microsize iridium tensile test specimens.

2.1 TASK 1 - PLUTONIA/CURIA COMPATIBILITY AND BCL-PMC STABILITY

Microprobe examination was completed on two plutonia/rhenium specimens. An original plutonia (~1 w/o metallic impurities) specimen exposed at 1800°C for 18,000 hours and a purer plutonia (~0.23 w/o metallic impurities) specimen exposed at 1800°C for 15,000 hours exhibited identical results. Rhenium mass transported to the sidewalls of the capsule as essentially pure rhenium with other elements from the plutonia such as iron, chromium, and nickel below limits of detection (0.1% to 1%). Oxide-appearing inclusions in the mass transported rhenium were high in plutonium with Re, Si, Fe, or Ni below detection limits. This confirms results obtained on the short-term tests as previously reported (Reference 1). The major significance of rhenium transport is that material is removed from hot areas and transported to colder areas. Plutonia also moves in a similar manner but in doing so reduces the thermal gradient thereby reducing and eventually eliminating further transport.

2.2 TASK 2 - PMC AND SSC TESTING PROGRAM

Microprobe analysis has been completed on one PMC and one SSC specimen. Specimen No. PU270 (PMC/TZM/Mo-47Re, 1800°C, 5000 hr) exhibited large precipitates containing primarily titanium with lesser amounts of zirconium at the TZM/Mo-46Re interface. Small precipitates also present contained titanium, with the zirconium content apparently too small for detection. The

titanium content in this TZM is 4000 ppm; the zirconium was 500 ppm. No plutonium was detected at or near the surface of the TZM. The outer surface of the Mo-46Re, which was contained in a Ta-10W guard capsule, had a large number of holes and was high in tantalum. Presence of this level of precipitation in the TZM is indicative of oxygen transport through the alloy. Diffusion of oxygen continued through the Mo-46Re, but no alloying ingredients were present to produce precipitates from the permeating oxygen.

Specimen No. PU263 (SSC/TZM T-111, 1800°C, 5000 hr) exhibited hafnium-oxide precipitates in both the T-111 and TZM. The T-111 initially contained 2% hafnium, but the TZM started with none. Precipitates in the T-111 were essentially pure hafnia. The very fine precipitates in the TZM were a mixed oxide of hafnia apparently diffused from the T-111, and titania from the TZM. There were large hafnia precipitates at the PMC/TZM interface. Small plutonia particles were present at the TZM interface, but there was no plutonia diffusion into the TZM. As described in quarterly report No. 16 (Reference 2), the more volatile elements diffuse from one alloy to another at 1800°C. It is apparent that precipitation of hafnia-based oxides occurred in the TZM, but it is unlikely that this precipitation was harmful to its function as a liner or compatibility barrier.

2.3 TASK 3 - MHW/RTG BARRIER TEST

No experimental effort was expended on the microprobe specimens during the current quarter. This is the only work remaining to complete this task.

2.4 TASK 4 - PLUTONIA FUEL AGING

Experimental effort was completed in the previous quarter with only the topical report remaining to conclude this task.

2.5 TASK 5 - MHW/RTG MOCKUP TESTS

Objectives of the MHW mockup tests are to determine iridium mechanical behavior, vent performance, and the possible presence of detrimental materials interactions, using systems oriented test configurations. A comprehensive discussion of the various configurations, test procedure, and assembly preparations was presented in quarterly report No. 16 (Reference 2). Figures 2-1 and 2-2 show the test assembly configurations.

2.5.1 Compatibility Results

Exposure was completed in early December on the first set of specimens (Table 2-1). Macroscopic observations are listed in Table 2-2, vent flow data in Table 2-3, plutonia fuel dimensional data in Table 2-4, and microscopic observations in Table 2-5.

One of the fueled test specimen vents plugged, one partially plugged, and one was unaffected by exposure. The vent in M-1 (uniform temperature, 1250 hr) was plugged as determined by post-test flow measurement. The presence of 20 grams of coarse plutonia powder in M-1, with a high surface area, resulted in transfer of plutonia into the general area of the vent, with visible particles between the capsule lid and vent cover. Although it could have contained plutonia, no plutonia was observed in the vent. The vent was almost ground through in polishing (A, Figure 2-3). The reason for almost missing the vent hole entirely is that the vent is probably plugged with iridium transported from either the vent cover or the iridium powder between the lid and the vent cover. There are plutonia particles between the lid and vent cover, as shown by the gray particle in B, Figure 2-3. Almost all of the iridium powder is missing between the vent cover and lid which could have provided a source for transport. There is also a reaction layer visible on the outer surface of the vent area as shown in C, Figure 2-3.

The vent in specimen No. T-1 (thermal gradient, 1250-hr) had flow reduced from 103 to 8.2 cc/hr. As shown in both the unetched and etched photomicrographs in Figure 2-4, the core of the vent appears to be mass-transported iridium. The central area of the vent cover appears to have less iridium powder remaining than in the peripheral area. There does not appear to be plutonia inside the vent, but plutonia is present between the lid and vent cover. The vent in specimen No. T-3 (thermal gradient, 1250 hr and 5 min at 1950°C) showed no difference between pre-test and post-test flow. There was no observable plugging of the vent, as shown in Figure 2-5. The hole appears as clean as that of control specimen No. C-1 (thermal gradient, 1250 hr) shown in Figure 2-6, except that the T-3 vent has not been sectioned quite to the centerline. The delamination of the iridium by the electron-beam drilling is clearly shown in both Figures 2-5 and 2-6, independent of the presence of plutonia. There were plutonia particles between the lid and vent cover of specimen No. T-3.

Figure 2-1. T Series Thermal Gradient (1440°C at bottom, 1100°C at top)

Figure 2-2. M Series Uniform Temperature (1440°C)

Table 2-1

MHW-RTG MOCKUP TEST SPECIMEN STATUS

Test Number	Fuel Form	Tensile Specimen	Vent Flow		Exposure Conditions		Reentry Thermal Pulse	Status (1-1-73)
			Pre Test (cc/hr)	Post Test (cc/hr)	Time (hr)	Temperature (°C)		
T-1	Pellet	DWDL Micro	103	8.2	1250	1440/1100	No	Examined
C-1	None	↓	---	---	1250	↓	No	Examined
T-2	Pellet		199		Optional (c)		No	1320 hr
T-3	Pellet		47	45	1250		Yes	Examined
C-3	None		193b	153	1250		Yes	Examined
T-4	Pellet		13		Optional (c)		Yes	1320 hr
C-4	None		80		Optional (c)		Yes	1320 hr
M-1	Powder	General Electric Subsize	145	<0.05	1250	1440	No	Examined
MC-1	None	↓	(a)	(a)	1250	↓	No	Examined
M-3	Powder		256		Optional		Yes	1320 hr
MC-3	None		(a)		Optional		Yes	1320 hr
M-4	Powder		700		Optional (c)		Yes	1320 hr
MC-4	None		157b		Optional (c)		Yes	1320 hr

a. No iridium inner capsule.

b. No cover on iridium inner capsule.

c. 5000 hr unless changed by MHW program requirements.

Table 2-2
MACROSCOPIC OBSERVATIONS

Specimen Number	Plutonia Fuel	Iridium Tensile Specimens	Iridium Inner Capsule			Graphite		Iridium Outer Capsule or Foil
			Inside	Outside	Vent Area	Impregnated ThorneI-50	POCO and ATJ	
T-1	Slightly shadowed where tensile specimens located. Rest of appearance like before test.	Clean where in contact with fuel, slightly gray above fuel. Backside of specimens very clean with gage length distortion.	Bottom looked good. Sidewalls next to fuel had gray color. Bright next to retainer. 3-mil retainer bent ~180° ~1T before breaking.	Top had etched appearance. Sidewalls were clean with minor shadowing.	Inside cover had black adherent deposit. Top was clean.	Undamaged with slightly different appearance opposite vent hole.	Excellent appearance.	Inside had shadowing from graphite. Outside was gray.
C-1	No fuel.	Dark gray discolorations.	Bright and shiny.	Shadowed.	Clean (no vent cover).	Excellent appearance, no delamination. Faint gold color.	No degradation. Gold color on surface.	Foil was shadowed adjacent to graphite. Very ductile especially at hot end. No self welding.
T-3	Less shadowing from tensile specimens than T-1, chipped corner. As received. appearance.	Same as T-1.	Bottom was shadowed by fuel. Sidewalls like T-1. 3-mil retainer bent 180°, ~1T before breaking.	Clean. Minor shadowing.	Inside cover light gray. Portions of Ir powder missing.	Spot opposite vent hole (~1/8-in.) that did not appear to be a depression.	Same as T-1.	Inside had shadowing from graphite. Outside was very bright.
C-3	No fuel.	Gold interference colors on 2 of 4 specimens.	Bright and shiny.	Bright and shiny.	Same as C-1.	Same as C-1 with minor delamination.	Same as C-1.	Brighter than C-1. Self welding Ductile but less than C-1.
M-1	Slight sintering of powder, broke up easily on prodding.	Discoloration above fuel level. Loosely adherent powder on surface in buried area rubbed off with stick.	More shadowing than T-1 and T-3.	Clean except for black deposits on lid.	Black deposits on bottom and top of vent cover (split apart from lid), appeared plugged with black speck on outside.	Appears eroded opposite vent hole (1/8-in. dia and 15 mil deep).	Same as T-1	Inside had shadowing from graphite. Outside was deeper gray and rougher than T-1.
MC-1	No fuel.	Excellent appearance very bright.	3-mil foil was clean and bright.	3-mil foil was clean and bright.	No vent.	Delamination.	Same as C-1.	Foil had modest shadowing adjacent to graphite. Self welding. Same ductility as C-3.

Table 2-3
VENT FLOW DATA*

Specimen Number	Pre-Test Flow (cc/hr)	Post-Test Flow (cc/hr)
T-1	103	8.2
C-1	Not measured	Not measured
T-3	47	45
C-3**	193	153
M-1	145	Plugged (<0.05)
MC-1	No capsule	No capsule

*Test performed with a pressure differential of one atmosphere of air. to vacuum. Accuracy of pre-test flow was $\pm 2\%$ of the recorded value; post-test accuracy was $+3\%$.

**No vent cover.

Table 2-4
PLUTONIA FUEL DIMENSION DATA

Specimen Number	Pre-Test			Post-Test		
	Length (in.)	Diameter (in.)	Across Flats (in.)	Length (in.)	Diameter (in.)	Across Flats (in.)
T-1	0.6466	0.489	0.3545	0.645	0.486- 0.488	0.353- 0.354
T-3	0.6531	0.4888	0.3556	0.652- 0.654	0.489- 0.490	0.355

Table 2-5
MICROSCOPIC OBSERVATIONS

Specimen Number	Plutonia Fuel	Iridium Capsule	Vent Area
T-1	Single phase	Bottom has reaction layer with voids at interface with the unattacked material (0.5 mil thick). Reaction layer goes up side wall tapering off toward the top. Fine grain structure.	Vent almost plugged with iridium. Delaminations in vent hole. Plutonia between lid and vent cover.
C-1	No plutonia	Fine grain structure. No attack.	Delamination in vent hole. No deposits or attack. No vent cover.
T-3	Single phase except for small quantity of gray second phase in grain boundaries	String of pores along bottom and up sidewalls which appears inter-metallic when etched (0.4 mil thick).	Plutonia between lid and vent cover. Bottom of vent bridged over-(not quite ground to centerline). Unknown material on outside of vent exit.
C-3	No plutonia	Coarse grain structure. No attack.	Delamination in vent hole. No deposits or attack. No vent cover.
M-1	Plutonia powder not examined	Small pores on bottom of inside, localized, not on wall to depth of 0.1 mil. (Capsule was not in direct contact with plutonia because an iridium sub-capsule was used to hold the sub-size tensile specimens).	Plutonia between lid and vent cover. Ground past centerline of vent hole; unable to determine reason for plugging. Reaction (0.5 mil thick) on outside at vent area.
MC-1	No plutonia	No capsule.	No vent.

Figure 2-3. Specimen No. M-1 Vent Area

A. LID WITH VENT (TOP) AND VENT COVER (BOTTOM), UNETCHED

B. LID WITH VENT (TOP) AND VENT COVER (BOTTOM), ETCHED

Figure 2-4. Specimen No T-1 Vent Area

A. LID WITH VENT (TOP) AND
VENT COVER (BOTTOM), UNETCHED

50x (20 MILS)

B. LID WITH VENT (TOP) AND
VENT COVER (BOTTOM), ETCHED

50x (20 MILS)

C. VENT AREA SHOWING DELAMINATIONS, ETCHED

100x (10 MILS)

Figure 2-5. Specimen No. T-3 Vent Area

Figure 2-6. Specimen No. C-1 Vent Area

Iridium capsules reacted with tungsten present in the plutonia but not with the plutonia itself or the external graphite. The largest reaction (~ 0.5 mil deep) was observed on the bottom and up the wall of specimen No. T-1 as shown in Figure 2-7. The T-1 reaction layer was continuous and was accompanied by a layer of voids. Both specimen No. T-3 and M-1 also had strings of fine pores very close to the inner surface but no visual reaction layer as such (Figures 2-8 and 2-9). The presence of this layer was unexpected in specimen No. M-1 inasmuch as there was no physical contact between the plutonia and the iridium capsule. The iridium exposed only at $\sim 1370^{\circ}\text{C}$ exhibited a fine grain size, but the specimens that underwent the 5-min-at- 1950°C thermal pulse exhibited grain growth to the point that major areas of the wall had only 2 to 3 grains across the wall as visible in the etched photomicrographs.

Plutonia remained in very good condition as shown in the macrograph in Figure 2-10. Dimensional measurement showed very slight shrinkage, almost within limits of measurement for specimen No. T-1 and no change for specimen No. T-3. Visually, there, was no change in both pellets.

A. BOTTOM, UNETCHED

500x (2 MILS)

B. BOTTOM, ETCHED

500x (2 MILS)

C. SIDE WALL NEAR TOP OF FUEL, ETCHED

500x (2 MILS)

Figure 2-7. Specimen No. T-1 Iridium Capsule Reaction Areas

A. BOTTOM, UNETCHED

500x (2 MILS)

B. BOTTOM, ETCHED

500x (2 MILS)

Figure 2-8. Specimen No. T-3 Iridium Capsule Reaction Area

BOTTOM, ETCHED

500x (2 MILS)

Figure 2-9. Specimen No. M-1 Iridium Capsule Reaction Area

Figure 2-10. Post-Test Macrographs of Plutonia, T-1 (left), T-3 (right)

Microscopically, both the T-1 and T-3 pellets showed some coalescence of porosity and a basically single-phase structure. It is probable that some reduction in oxygen occurred during exposure but that reoxidation occurred on handling in air afterwards. The T-3 specimen had small areas of gray phase associated with grain boundaries as shown in Figure 2-11. This phase has been seen previously in plutonia supplied by LASL but remains unidentified. It is not an artifact. Handling and polishing were performed with organic-free materials to eliminate the possibility of reaction products. Mounted pellets and the remainder of the plutonia pellets have been sent to LASL for chemical and microprobe analyses. Chemical analysis will be made to determine whether changes in composition occurred during exposure. Microprobe evaluation may be able to determine composition of the presently unidentified gray phase. The graphites exhibited no structural damage, other than minor erosion opposite the vent hole of the impregnated Thornel-50, as shown in Figure 2-12. The impregnated Thornel-50 graphite in specimen No. M-1 graphite had a definitely eroded area 0.125 in. diameter by 0.015 in. deep area, indicating that the vent was open for at least some time before plugging. Several of the ATJ and POCO-AXF-5Q graphite pieces, particularly those exposed in the control specimens, had gold-colored surface areas. This color is believed to be a tungsten carbide, partially caused during vacuum outgassing which utilized tungsten heating elements, and from volatilization of tungsten from the tungsten guard capsules surrounding the graphite. Iridium foil prevented direct contact but did not eliminate vapor contact.

2.5.2 Mechanical Testing Results

Tensile specimens had the same general appearance as the capsules except for spots of either gold or matte-gray areas in the control specimens and loosely adhered plutonia in the M-1 specimens (Figure 2-13). Photographs taken of the reaction areas in the control specimens show both a gold-colored thick deposit and matte-gray or bubbled areas (Figure 2-14). Both have been determined to be platinum by x-ray techniques. Source of the platinum is unknown. The loosely adhered plutonia was partially removed using wooden sticks as scrubbing tools. All the fueled test tensile specimens were cleaned ultrasonically in water, in a variety of solutions from pure water, to soap solutions, to nitric acid. Surfaces remained highly contaminated at greater than 100,000 d/m smear count. The M-1 specimens and MC-1 subsize

Figure 2-11. Specimen No. T-3 Plutonia

Figure 2-12. Post-Test Macrographs of Impregnated Thornel-50 Graphite, T-1 (left), T-3 (center), M-1 (right)

Figure 2-13. Macrograph of M-1 Subsize Tensile Specimens

THICK GOLD COLORED AREA (TOP) AND
THIN GRAY MATTE AREA (TOWARD RIGHT)

20x (50 MILS)

Figure 2-14. Macrographs of Control Micro-Size Tensile Specimen C-1-CG-1

specimens have been stored, waiting GE instructions to ship to their testing facility. The T-1, T-3, C-1, and C-3 microsize tensile specimens have been tested, except for one specimen adjacent to the plutonia (inside the inner iridium capsule) and one specimen adjacent to the hot graphite in each of four test assemblies.

The high-strain-rate tensile testing device functioned almost perfectly in testing the iridium microtensile specimens. Load time traces and elongation measurements have been obtained on 7 pre-test, 14 post-test control, and 16 post-test fueled iridium tensile specimens. Specific test data are listed in Table 2-6; averaged data are listed in Table 2-7. The pre-test data for iridium annealed at 1500°C for 1 hr showed an average ultimate tensile strength of 40,000 psi (40 ksi) and average elongation of 58% (based on an effective gage length of 0.190 in.) when tested at 1400°C. Careful measurement of scribed specimen elongation has demonstrated that the effective gage length of the microtensile specimen is 0.190 in. at 1320°C. The 58% elongation appears valid, based on four specimens, and is somewhat higher than that reported by GE (47% average and a range from 44% to 50% at a strain rate of 4000 min⁻¹ and at 1380°C). The 40,000-psi tensile strength is somewhat higher than that reported by GE (28,200 average and a range from 26,800 to 30,100 at a strain rate of 4000 min⁻¹ and approximately 1380°C). This higher strength measurement may be inherent in this iridium lot which is high in tungsten content (0.8% W) or may be caused by test technique or specimen configuration. Tungsten has caused some degree of strengthening, but further testing is underway on several standard engineering materials to ensure test validity.

Tensile data on post-test specimens showed general decrease in elongation, from 58% to about 49%, and no general change in measured ultimate tensile strength for specimens aged at approximately 1370°C. Statistically there was no difference between fueled and control specimens but the aging treatment was significant at the 95% confidence level. The T-1 and C-1 specimens exhibited fine grain structures whereas the T-3 and C-3 specimens were coarse grained from the reentry thermal pulse.

The only exceptions to these generalizations were reductions in strength to 34-35 ksi in the C-3 (control, thermal gradient, ~1370°C, 1250 hr plus 5 min at 1950°C) specimens and reduced ductility in the specimens exposed in the colder graphite (~1200°C). Reduction in strength was statistically

Table 2-6

IRIDIUM MICROTENSILE TEST DATA

Specimen No.	Temp (°C)	Tensile Strength (ksi)	Elongation (b) (%)	Average Strain Rate (c) (10 ³ in./in.-min)	Specimen No. (a)	Temp (°C)	Tensile Strength (ksi)	Elongation (b) (%)	Average Strain Rate (c) (10 ³ in./in.-min)
<u>Pre-Test</u>									
1 Notch	1320	44	52	22					
2 Notch	1320	46	57	22					
P-1	1390	34	57	19					
P-2	1400	42	63	23					
P-3	1400	40	55	21					
P-4	1395	42	55	21					
P-5	1350 (g)	47	58	21					
<u>Post-Test</u>					<u>Post-Test</u>				
C-1-I-1	1365	37	47	19	T-1-I-1	1405	47	48	11 (f)
C-1-I-2	1395	39	49	20	T-1-I-2	1400	43	51	20
C-1-I-3	1400	38	54	20	T-1-I-3	1400	39	47	21
C-1-HG-1	1400	45	47	18	T-1-HG-1	1395	37	56	20
C-1-HG-2	1390	38	46	19	T-1-HG-2	1395	39	51	18
C-1-HG-3	1400	36	44	16	T-1-HG-3	1405	36	43	18
C-1-CG-2	1400	40	33 (e)	14	T-1-CG-1	1405	39	58	23
					T-1-CG-2	1395	46	54	22
C-3-I-1	1385	36	49	19	T-3-I-1	1405	38	53	20
C-3-I-2	1385	36	47	20	T-3-I-2	1395	44	48	21
C-3-I-3	1405	31	53	19	T-3-I-3	1395	44	54	22
C-3-HG-1	1400	38	46	20	T-3-HG-1	1395	40	51	19
C-3-HG-2	1400	33	32	14	T-3-HG-2	1410	38	49	20
C-3-HG-3	1385	35	51	19	T-3-HG-3	1390	41	52	22
C-3-CG-2	1395	37	24 (d)	13	T-3-CG-1	1400	46	53	13 (f)
					T-3-CG-2	1405	35	24 (d)	6

- a. Specimen designations are: T-1 fueled, 1250 hr; C-1 control, 1250 hr
T-3 fueled, 1250 hr + 5 min at 1950°C, C-3 Control 1250 hr + 5 min at 1950°C
I, inside iridium capsule at 1370°C
HG, Outside iridium capsule adjacent to ATJ graphite at 1400°C
CG, In POCO graphite at 1200°C
P, Pretest - 1500°C, 1 hr
- b. Estimated effective gage length of 0.190 in. used for all elongation measurements.
- c. Average strain rate based on specimen elongation, effective gage length, and time to break.
- d. Broke near end of gage section.
- e. Broke at pin.
- f. Rubber bumper stuck to bottom of weight causing slower strain rate buildup.
- g. Specimen held 3 min at temperature; all other specimens held 10 to 30 sec.

Table 2-7
IRIDIUM MICROTENSILE TEST DATA AVERAGES

Specimen Group	Number of Specimens	Tensile Strength (ksi)	Elongation (%)	Comments
Pretest	2	45	54	1320°C
Pretest	4	40 ^(a)	58 ^(b)	1400°C (same as post-test)
C-1-I	3	38	50	
C-1-HG	3	40	46	
C-1-CG	1	40	33	Broke at pin.
T-1-I	3	43	49	
T-1-HG	3	37	50	
T-1-CG	2	42	56	
C-3-I	3	34	50	Lowered strength.
C-3-HG	3	35	43	Lowered strength; one specimen had low ductility.
C-3-CG	1	37	24	Broke near end of gage section.
T-3-I	3	42	52	
T-3-HG	3	40	51	
T-3-CG	2	40	39	One specimen broke near end of gage section.

a. Mean = 39.6; s = 3.5.

b. Mean = 57.5; s = 3.6.

significant; the cause is unknown. Three of the latter specimens broke near the end of the gage length or at the loading pin, probably indicating an inability of the material to deform in a ductile manner at the lower testing temperatures ($\sim 1000^{\circ}\text{C}$) in those locations.

In two scribed pretest specimens used to determine effective gage length, the region near the end of the gage length deformed less than 10% compared to over 50% in the hot central region. This reduced elongation is logical because of the increased strength at the end of the gage length where the temperature is between 1000° and 1100°C . Preliminary observations of these fractured specimens indicate brittle behavior in the cold regions showing less than the expected 10% local deformation.

The most valuable information from tensile testing is that exposure of iridium in contact with either plutonia or graphite at a typical MHW operating temperature without or with the reentry thermal pulse caused no degradation in either ductility or tensile strength compared to changes produced by thermal aging alone.

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Section 3

CONCLUSIONS

Rhenium and plutonia appear to be mutually insoluble at 1800°C, based on microprobe results. Temperature gradient mass transfer of both rhenium and plutonia occurs in closed systems at 1800°C but independent of each other. There was no evidence of any form of attack by plutonia on the container. It is believed that the presence of the oxygen partial pressure over the plutonia is responsible for the mass transport of rhenium from hot to cold surfaces.

Both titanium and hafnium diffuse from alloys containing these alloying constituents to those alloys not containing them when held together at 1800°C with TZM. Depth of precipitation was shallow enough not to have caused major degradation of the Mo-46Re bulk properties. Hafnium moved from T-111 into TZM causing massive precipitates but again, without any major bulk damage. This form of transport did not occur at 1500°C and, although important for thermionic devices, has little or no relevance to thermoelectric systems currently in use or under development.

MHW mockup tests have shown that vent plugging was caused by iridium transport but that elevated temperature mechanical properties of the iridium container remained unaffected by the presence of plutonia, graphite, and a thermal gradient.

Metallography has shown that an almost-plugged vent was filled with iridium. The source of the iridium was most likely the iridium powder on the vent cover which was at a slightly higher temperature than the vent. The presence of a small but finite amount of oxygen vapor species from the plutonia aided the thermal gradient in providing the driving force for transport. Iridium as a potential source of oxygen was minimized by acid cleaning, vacuum heat treatment, and vacuum electron-beam welding used for assembly. Either enlarging the vent hole or a different configuration appears to be required to prevent plugging of the MHW fuel sphere assembly.

Iridium and plutonia are chemically compatible with no interaction present. However, tungsten present in the plutonia created a tungsten-iridium inter-metallic layer less than one mil thick. This layer did not affect the bulk mechanical properties of the iridium, based on tensile test data.

Iridium mechanical behavior appears to be only slightly degraded by service at typical MHW heat source operating temperatures. Presence of plutonia had no affect beyond that of the thermal treatment alone. Limited test results indicates that there is a probability that thermal treatment at lower temperatures, such as would exist during heat source storage or on the launch pad, could cause embrittlement of the iridium if impact were to occur at temperatures in the vicinity of 1000°C. Further testing appears essential to determine whether this may cause sufficiently low ductility to result in breaching upon impact.

Section 4
REFERENCES

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