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Flight-Systems Safety Program

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FLIGHT SYSTEMS SAFETY PROGRAM

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ABSTRACT

This technical monthly report covers studies related to the use of $^{238}\text{PuO}_2$ in radioisotope power systems carried out for the Office of Coordination and Special Projects of the US Department of Energy by Los Alamos National Laboratory.

Most of the studies discussed here are ongoing. Results and conclusions described may change as the work continues. Published reference of the results cited in this report should not be made without the explicit permission of the person in charge of the work.

I. GENERAL-PURPOSE HEAT SOURCE (GPHS)

A. Post-Mortem Analysis (F. W. Schonfeld)

The chemical analyses of the iridium cups used in the second design iteration test, DIT-2, were completed, and the microscopic examinations of the DIT-2 fuel pellets were finished.

Results of the spectrographic analyses of the eight cups used to make the four fueled clads used in the DIT-2 impact are listed in Table I. Although the cups were made from several iridium powder batches and different sheets from each powder batch, the reproducibility of the chemical compositions is good.

The compositions of the grain boundaries of the eight cups were determined by Auger electron spectroscopy (AES) of freshly formed fracture surfaces and are reported in Table II. With the exception of the vented cup from SR-106, the variations in thorium intensities are normal for DOP-26 iridium not exposed for long times to PuO_2 . Some of the samples analyzed contained silicon, phosphorus, or sulfur.

All samples examined had oxygen and carbon at the grain boundaries. There is a general tendency to ignore

these two elements because they can be condensed from the residual gases present in the vacuum system used for AES. If they were present in the iridium and not merely condensed on the surface after fracture, alternative explanations of some phenomena observed in the DOP-26 iridium welds could be advanced. For example, the cracks in the weld overlap might occur because of carbon, not thorium, segregation to the weld center line. Similarly, the gas porosity sometimes found in the first-pass metal in the weld overlap may result from oxygen diffusion to and reaction with graphite flakes, which constitute one component of the iridium-carbon eutectic that precipitated near the center line during the first weld pass.

The gross failure modes of the four fuel pellets can be seen in Fig. 1. X-radiographic examinations showed only SR-106 to contain "large cracks" prior to heat treating and impacting. The prime pellets (SR-107 and SR-110) appear to have fragmented more than the other pair. The fuel fractures were entirely brittle.

The microstructures of the four fuel pellets are shown in Figs. 2 through 5. There are microstructural variations among the pellets and within each pellet. Such variations are inevitable, given the small batch process that must be

TABLE I. Spectrographic Analyses^a of GPHS Clads Used in DIT-2

Element	SR-105		SR-106		SR-107		SR-110	
	1 (MER-2) V-cup ^b	2 (N-505-6) WS-cup ^c	3 (N 504-5) V-cup	4 (MERR 3-3) WS-cup	5 (LR 300-2) V-cup	6 (MR-432-2) WS-cup	7 (N 515-3) V-cup	8 (NR 576-6) WS-cup
Li	<100	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Be	<3	<DL	<DL	<DL	<DL	<DL	<DL	<DL
B	<10	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Na	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Mg	25	20	15	3	3	3	15	15
Al	40	50	40	40	40	40	70	50
Si	20	20	20	10	10	10	20	20
P	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
K	<3000	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ca	3	<3	<DL	<DL	<DL	<DL	<DL	<DL
Ti	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
V	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Cr	10	<10	<DL	<DL	<DL	<DL	10	10
Mn	<10	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Fe	40	20	20	30	30	30	100	80
Co	<10	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ni	20	10	<10	20	20	20	50	40
Cu	20	15	5	15	15	15	20	15
Zn	<50	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ga	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ge	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Sr	<3	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Zr	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Nb	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Mo	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ru	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Rh	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Pd	<20	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ag	<10	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Cd	<100	<DL	<DL	<DL	<DL	<DL	<DL	<DL
In	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Sn	<50	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Sb	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ba	<3	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Hf	<300	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Ta	<1000	<DL	<DL	<DL	<DL	<DL	<DL	<DL
W	0.1-1%	0.1-1%	0.1-1%	0.1-1%	0.1-1%	0.1-1%	0.1-1%	0.1-1%
Pt	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Au	<10	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Tl	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Pb	<50	<DL	<DL	<DL	<DL	<DL	<DL	<DL
Bi	<30	<DL	<DL	<DL	<DL	<DL	<DL	<DL

^aResult preceded by < symbol represents limit of detection. To increase clarity, detection limits are indicated in column 1, column 2 (Ca) and (Cr), and column 3 (Ni); <DL means less than detection limit.

^bThe cup containing the vent.

^cThe cup containing the weld shield.

TABLE II. Auger Intensity Ratios from Wall Samples of GPHS Clads Used in DIT-2

Capsule	Th ₆₅ /Ir ₂₂₉			Th ₆₅ /Ir ₂₂₉			Si ₉₂ /Ir ₂₂₉			P ₁₂₀ /Ir ₂₂₉			S ₁₅₀ /Ir ₂₂₉			C ₂₇₀ /Ir ₂₉₉			O ₅₁₀ /Ir ₂₂₉		
	in.	cen.	out.	in.	cen.	out.	in.	cen.	out.	in.	cen.	out.	in.	cen.	out.	in.	cen.	out.	in.	cen.	out.
SR-105																					
V cup (1)	0.078	0.069	0.064	0.580	0.500	0.444	---	---	---	---	---	---	---	---	---	0.420	0.448	0.478	---	---	0.622
(2)	---	0.075	---	---	0.509	---	---	---	---	---	---	---	---	---	---	---	0.237	---	---	---	---
WS cup (1)	0.059	0.053	0.069	0.407	0.412	0.439	---	---	0.018	---	---	---	---	---	0.070	0.833	0.708	1.149	0.806	0.888	0.877
(2)	---	0.057	---	---	0.440	---	---	---	---	---	---	---	---	---	---	---	0.510	---	---	0.820	---
sputtered	---	---	0.003	---	---	0.020	---	---	---	---	---	---	---	---	---	---	---	0.570	---	---	---
SR-106																					
V cup (1)	0.018	0.046	0.059	0.135	0.381	0.429	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
(2)	---	0.044	---	---	0.347	---	---	---	---	---	---	---	---	---	---	NO	NO	NO	NO	NO	NO
WS cup (1)	0.074	0.090	0.068	0.483	0.652	0.469	---	---	---	---	---	---	---	---	---	0.367	0.717	0.837	0.475	0.435	0.480
(2)	---	0.077	---	---	0.566	---	---	---	---	---	---	---	---	---	---	---	0.462	---	---	0.453	---
SR-107																					
V cup (1)	0.048	0.008	0.062	0.346	0.059	0.418	---	---	---	---	---	---	---	0.025	0.155	0.385	0.490	0.755	0.664	0.363	0.964
(2)	---	0.005	---	---	0.039	---	---	---	---	---	---	---	---	---	---	---	0.481	---	---	0.433	---
sputtered	---	---	Tr	---	---	Tr	---	---	---	---	---	---	---	0.019	---	---	0.606	---	---	---	---
WS cup (1)	0.053	0.052	0.057	0.364	0.357	0.408	---	---	---	---	---	---	---	---	---	---	0.606	---	---	---	---
(2)	---	0.072	---	---	0.533	---	---	---	---	---	---	0.097	0.109	0.119	0.039	0.491	0.536	0.602	0.782	0.952	0.728
sputtered	Tr	---	---	Tr	---	---	---	---	---	---	---	---	---	0.076	---	---	0.438	---	---	0.971	---
SR-110																					
V cup (1)	0.047	0.048	0.063	0.359	0.355	0.480	---	---	---	---	---	0.100	---	0.027	---	0.576	0.518	0.350	0.755	0.800	0.800
(2)	---	0.057	---	---	0.418	---	---	---	---	---	---	---	---	---	---	---	0.764	---	---	0.709	---
WS cup (1)	0.072	0.073	0.051	0.528	0.537	0.308	---	---	0.308	---	---	---	---	0.031	0.307	---	0.367	0.539	---	---	0.862
(2)	---	0.063	---	---	0.467	---	---	0.168	---	---	0.112	---	---	---	---	---	---	0.206	---	0.299	---
sputtered	Tr	---	---	---	---	Tr	---	---	---	---	---	---	---	---	---	---	---	0.562	---	---	---

(a)

Fig. 1. The DIT fuel pellets all failed in a brittle manner.
(a) SR-105, (b) SR-106, (c) SR-107, and (d) SR-108, 1.5X.

(b)

(c)

Fig. 1. (cont)

(d)

(a)

(b)

Fig. 2. The microstructures of center and edge regions from fuel pellet SR-105 were similar. (a) Center and (b) edge, 50X.

(a)

(b)

Fig. 3. The central region of fuel pellet SR-106 was more porous than the edge. (a) Center and (b) edge, 50X.

(a)

(b)

Fig. 4. The exterior region of fuel pellets SR-107 had a less pronounced shard-like microstructure than the edge. (a) Center and (b) edge, 50X.

(a)

(b)

Fig. 5. The interior of fuel pellet SR-110 was more homogeneous than the exterior. (a) Center and (b) edge, 50X.

used to make the pellets. The average diametral height strain sustained by the fueled clads in the DIT-2 impact ranged from -4.2% (SR-105) to -6.5% (SR-110). How much of this difference should be attributed to microstructural differences and how much to gross cracks is unknown.

The impacted fuel from SR-110 was recovered underwater and subjected to a size analysis. The results are given in Table III. Both the -10- μ m fraction and the $\leq$ 4- μ m fraction are smaller than in pellet SR-48, analyzed after the DIT-1 impact. (-10 μ m = 0.0022 and $\leq$ 4 μ m = 0.0002.)

Samples of each fuel pellet were analyzed chemically, and the results are reported in Table IV. Three of the pellets had more boron and magnesium than allowed in the shards from which the pellets are pressed, and two pellets also contained excess silicon.¹

B. Impact Tests (R. W. Zocher)

The test plan for the DIT-3 impact was issued.² The test objectives are to impact the module with the impact shell axes at 30° to the target and to determine if fueled

TABLE III. Sieve Analysis of the Fuel from Capsule SR-110 After Impact at 58 m/s and 930°C

Particle Size (Microns)	Weight Fraction	Accumulated Weight Fraction
+6000	0.4620	0.4620
+2000	0.3463	0.8083
+841	0.1269	0.8352
+420	0.0304	0.9656
+177	0.0125	0.9781
+125	0.0027	0.9808
+74	0.0042	0.9850
+45	0.0031	0.9880
+30	0.0043	0.9922
+20	0.0024	0.9947
+10	0.0044	0.9991
-10	0.0009	1.0000
$\leq$ 4	0.0001	

TABLE IV. Chemical Analyses: Spectrographic Analyses of DIT-2 Fuel

Element	SR-105	SR-106	SR-107	SR-110
Li	1	1	2	2
Be	<1	<1	<1	<1
B	4 ^c	3 ^c	3 ^c	1
Na	5	2	7	2
Mg	100 ^c	100 ^c	100 ^c	20
Al	20	8	30	45
Si	230 ^c	180	240 ^c	200
K	<5	<5	<5	<5
Ca	250	100	300	150
Ti	150	100	100	60
V	<3	<3	<3	<3
Cr	40	40	30	80
Mn	10	7	8	4
Fe	340	250	300	410
Co	<3	<3	<3	<3
Ni	20	20	20	9
Cu	<1	<1	<1	<1
Zn	8	8	8	<5
Rb	<10	<10	<10	<10
Sr	<5	<5	<5	<5
Y	<25	<25	<25	<25
Zr	<100	<100	<100	<100
Nb	<10	<10	<10	<10
Mo	5	5	4	<3
Ag	10	10	30	5
Cd	<10	<10	<10	<10
Sn	<5	<5	<5	<5
Ba	7	<2	2	<2
La	<25	<25	<25	<25
Hf	<25	<25	<25	<25
Ta	1000	1000	500	100
W	<10	<10	<10	<10
Re	<25	<25	<25	<25
Pb	15	<5	<5	<5
Bi	<1	<1	<1	<1
P ^a	<20	<20	<20	<20
P ^b	25	30	40	25

^aBy radioanalysis.

^bBy wet-chemical analysis.

^cExceeds allowable level.

clads with weld-overlap cracks can be impacted without failing. The plan calls for impacting the module under conditions that would obtain in a minimum-gamma reentry, because those conditions are most conducive to the propagation of existing cracks.

C. Early Compatibility Test (D. Pavone)

The early compatibility test was initiated on February 18, 1982. The assembly consists of two GPHS fueled clads contained in a fine-weave pierced fabric (FWPF) graphic impact shell (GIS) and a set of carbon-based carbon fiber (CBCF) insulation held in an ATJ-graphite heat-treat fixture. The helium release from the assembly is being monitored continuously. The test temperature, as measured by sighting with an optical pyrometer on the end of one of the iridium clads through small holes in the ATJ fixture, CBCF insulation, and FWPF GIS, is 1310°C.

No evidence of vent plugging has been observed. The accumulated time of exposure as of March 1, 1982, was 256 h.

II. MULTI-HUNDRED WATT (D. Pavone)

Test assembly MHFT-73 was aged in vacuum for 8820 h at a graphite surface temperature of 1210°C, subjected to a simulated orbital-decay reentry pulse (1500°C peak temperature), and impacted at 1430°C and 81.6 m/s.

The cap of the GIS was oriented at 180° to the target face and the body was split longitudinally and delaminated. The iridium shell was oriented with the plane of the equatorial weld nearly perpendicular to the plane of the target. Four failures of the iridium clad were observed and are illustrated in Fig. 6. The most severe failure was a fuel push-through near the periphery of the impact face. The maximum width of this penetration was about 3 mm, and its length was about 20 mm. Additionally, a hoop crack about 1 mm wide by 10 mm long and a hoop-like crack 0.5 mm wide by 5 mm long near the vent assembly weld were observed. A reentrant fold about 25 mm long with a crack at the apex along the center line of the weld in the center of the impact face also was present. Visually, the edges of the push-through fracture appeared to have necked down, indicating a

(a)

(b)

Fig. 6. Photographs of the impacted iridium shell of MHFT-73. (a) Impact face showing the severe fuel push-through failure (~1X) and (b) profile view showing the hoop crack (~1X).

ductile fracture, whereas the other fractures appeared to be brittle.

III. HELIUM RELEASE (D. E. Peterson and J. S. Starzynski)

The helium released from eight additional samples of GPHS pellet GP-19 was measured this month. In these experiments, the samples were heated to an intermediate temperature at a rate of either 120 or 240°C/min, held at that temperature for 3 h, and then rapidly heated to 1600°C, where they were maintained until the helium signal had fallen to a constant background level. The details of the tests are given in Table V.

The logarithms of the effective diffusion constants vs the reciprocal temperatures are shown in Fig. 7. Runs 51 and 52 were linear over the entire temperature range so least-squares lines are shown in place of individual

points. Individual data points are plotted for runs 43-50, because these curves were not linear over their entire temperature ranges. Because conduction predominates in heat transfer at low temperatures and radiation becomes more important above 1000°C, it is possible that the tungsten cell, which has three times the thermal conductivity of the tantalum cell used for the other samples, conducted heat to the samples more efficiently. This is supported by the good agreement in the magnitude and temperature dependence of D' above 1000°C for most of the current and previous studies.

The least-squares fit for the results of an early experiment (09) at higher temperatures also agrees well with the current data. It appears that the helium release between 700 and ~1300°C can be modeled using a straight line like those for 51 or 52, but that at higher temperatures, it appears that there is an exponential increase in the diffusion constant (runs 21 and 24).

TABLE V. Test Conditions of Helium Release

Run No.	Sample Wt g	Age yr	Ramp Rate °C/min	Intermediate	Helium scc/g	Released %
				Temp °C		
43	0.835	0.764	120	900	0.367	91.4
45	1.100	0.769	120	1000	0.381	100.0
46	0.696	0.780	120	1000	0.366	89.8
47	0.745	0.783	240	1000	0.389	95.0
49	0.554	0.955	240	1200	0.385	92.5
50	0.805	0.818	240	1200	0.437	100.0
51 ^a	0.815	1.186	120	1200	0.464	75.0
52 ^a	0.797	1.186	120	1200	0.529	85.5

^aPerformed in tungsten container. All others were done in tantalum containers.

Fig. 7. Temperature dependence of the logarithm of effective diffusion constant D' for helium release from plutonia.

REFERENCES

1. "Specifications for General Purpose Heat Source Fuel Pellet Acceptance," Savannah River Plant document DPSP-79-1076, March 5, 1980.
2. R. W. Zocher, "GPHS Impact Test Plan—DIT-3," Los Alamos National Laboratory document CMB-5-C-82-6, February 8, 1982.

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