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Compiled by
S. E. Bronisz

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SPACE NUCLEAR SAFETY AND FUELS PROGRAM

September 1981

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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

A. Impact Test Plans (R. Zocher)

All efforts to complete the General-Purpose Heat Source (GPHS) half-module design iteration test (DIT)-1.5 were halted at the direction of the Department of Energy's Office of Coordination and Special Projects. In place of the cancelled test, we completed a full-module impact test, DIT-2. This test was a duplicate of DIT-1, except that the fueled clads had been welded by the Savannah River Plant (SRP) instead of by the Los Alamos National Laboratory and three thermocouples were used instead of one to monitor the clad temperature in each impact shell and on the aeroshell surface respectively.

B. DIT-2 Test Components (R. Zocher, F. Schonfeld)

1. **Reentry Shell.** The reentry shell used in the DIT-2 impact was fabricated at Los Alamos from flight-quality FWPF* graphite. A uniform 1.3-mm-thick layer was

machined from each exterior surface to simulate the ablation that would occur during the atmospheric reentry of a GPHS module. A 3-mm-deep, flat-bottomed, thermocouple hole was bored in the center of the back surface of the reentry shell, and two holes were drilled through the back wall in the locations of the centers of the fueled clads in the closure ends of the impact shells.

2. **Impact Shells.** The two graphite impact shells (GIS) (TGL-8 and TGL-10) used in the DIT-2 impact were fabricated from FWPF graphite by Los Alamos, and their chamfered corners were coated with pyrolytic graphite by the manufacturer, Pfizer, Inc.* A radial hole was drilled through each impact shell wall in a position corresponding to the center of the closure-end fueled clad.

3. **Insulation.** The carbon-bonded, carbon-fiber (CBCF) insulation used in the DIT-2 impact was supplied by the Oak Ridge National Laboratory (ORNL). The CBCF sleeves (C-27-6 and C-27-7) were from the same production lot used in the March thermal test.¹

*Fineweave-Pierced Fabric 3D carbon/carbon composite, a product of AVCO Systems Division, 201 Lowell St., Wilmington, MA 01887.

*Pfizer, Inc., P. O. Box 548, Easton, PA 18042.

months at 1210°C (GIS surface) in a vacuum. The iridium clad did not fail, but there were small, fingerprint-like cracks on its impact face.

The tantalum impact can was deformed somewhat unsymmetrically but was not breached. White smoke was observed issuing from the interior upon initial penetration of the can, and a small quantity of white material was deposited on the edges of the cut, as shown in Fig. 4.

The impact of the FSA occurred in a normal configuration with the cap of the GIS oriented at 180° to the initial point of impact. The GIS was delaminated, but its body was not split. A gray deposit was present on the interior of the GIS cap. Tantalum was the only element detected by electron microprobe analysis in this coating.

Figure 5 shows photographs of the fueled clad after it was removed from the GIS. The exterior surface of the iridium clad was a dull, medium gray, in contrast to the metallic appearance of previously examined assemblies. The gray coating was well bonded to the iridium. In addition, spots of yellow and rust-brown nonmetallic deposits were present on the impact face. No fractures of the iridium clad were observed, and only a few fingerprint-like cracks could be detected with the naked eye. However, examination at 7X magnification revealed a network of small cracks on the impact face. Irregular fuel fragment push-through deformation was present on the impact face, though the deformation of the iridium was not severe. The details of the test and summary results are compared to those other FSA impacts in Table I.

The gray surface coating of the iridium changed to a blue interference color on exposure to the laboratory atmosphere. Analysis of the surface indicated the presence of tantalum, iridium, and aluminum. Both the yellow and rust-brown nonmetallic deposits were found to contain tantalum, iridium, and silicon.

The grain size of the iridium clad was nonuniform. Very large grains were found in the interior two-thirds of the thickness, but much smaller grains were present on the remaining one-third. Typical microstructures are shown in Fig. 6. The statistical average grain sizes were 4.7 and 4.8 grains/thickness in the two hemispheres that make up the iridium clad. This grain size is somewhat smaller than that predicted from the data accumulated previously, but much coarser than that indicated by the ORNL grain-growth data. The results of Auger electron spectroscopy (AES) analysis of fracture surfaces of two samples of each hemisphere are presented in Table II and

summarized in Table III. These results show a depletion of thorium (particularly at the inside edge), high concentrations of silicon at a few locations, and a low concentration of phosphorous at one location. On an overall basis, the grain boundaries would be judged to be relatively clean. Emission spectrographic analysis of samples of each hemisphere indicated the impurities present in the iridium clad at levels greater than the detection limit were

Element	Hemisphere A	Hemisphere B
Aluminum	60 ppm	60 ppm
Silicon	30 ppm	15 ppm
Calcium	5 ppm	5 ppm
Cerium	10 ppm	15 ppm
Iron	300 ppm	300 ppm
Nickel	150 ppm	150 ppm
Copper	10 ppm	5 ppm
Platinum	30 ppm	30 ppm

Metallographic examination of samples of the plutonia sphere from the impact face (sphere surface) and the core did not reveal any abnormalities in microstructure or any impurity phases. Spectrographic analysis of a sample of the plutonia indicated the following elements to be present in amounts exceeding the detection limit.

Silicon	20 ppm	Zirconium	500 ppm
Zinc	5 ppm	Magnesium	7 ppm
Silver	10 ppm	Nickel	5 ppm
Calcium	750 ppm	Aluminum	60 ppm
Iron	75 ppm	Chromium	20 ppm
Titanium	200 ppm		

The quantities of calcium are above specification, and the amounts of titanium and zirconium are higher than those of other samples that have been analyzed.

Chemical analysis of the plutonia for phosphorus indicated 50 ppm. This value does not agree well with the pre-aging gamma-scan analysis of <20 ppm.

A rust-brown colored spot was observed on the surface of the plutonia sphere across from a corresponding deposit on the interior of the iridium clad. Sufficient material was scraped from the deposit for a qualitative spectrographic analysis, which showed the major constituent to be plutonium with detectable amounts of aluminum, titanium, silicon, calcium, cerium, manganese, iron, copper, silicon, and barium.

TABLE I. Impact Test Summary of Galileo Multi-Hundred Watt Fueled Sphere Assemblies

Assembly Identification	Aging Time (hours) 1210°C-GIS Vacuum	Impact Test Conditions		Post-Impact Dimensions			Failure Type				Type
		Reentry/Impact Temp (°C)	Velocity (m/s)	Height (mm)	Diameter (mm)	Per Cent Strain ^a Height/Diam	Hoop	Fingerprint ^b	Fuel Push-through	Other	
MHFT-61	8834	1550/1440	74	31.09	43.46	-23.5/+6.9			No Failures		DOP-26
MHFT-62	720	1550/1440	85	30.38	45.34	-25.6/+12.4	---	---	X	Weld bead back side	HDR
MHFT-64	720	1475/1300	85	29.03	45.11	-28.7/+10.8		(X)	---	---	DOP-26
MHFT-65	4320	1550/1440	85	28.55	43.84	-28.7/+8.0		(X)	X	If crack extends hoop area	
MHFT-66	720	1550/1440	85	30.68	43.64	-23.8/+6.9	---	X	---	---	HD
MHFT-67	100	1500/1430	74	29.64	43.66	-27.1/+7.4			No Failures		DOP-26
MHFT-68	110	1792/1579	82	30.07	43.66	-27.6/+7.4			No Failures		DOP-26
MHFT-69	100	--- /1037	82	30.99	43.71	-23.8/+7.6	---	(X)	---	Weld beam crack-IF	DOP-26
MHFT-70	720	1500/1430	82	29.16	43.26	-28.3/+6.4	---	X	X	---	DOP-26
MHFT-72	720	1500/1430	82	30.43	43.76	-25.1/+7.6			No Failures		DOP-26
MHFT-74	720	1500/1430	81	29.74	43.54	-26.8/+7.1			No Failures		DOP-26
MHFT-75	720	1500/1430	81	30.07	43.23	-26.0/+6.6-8.3			No Failures		DOP-26
					44.07						
MHFT-71	9215	1500/1430	82	29.79	42.98	-26.7/+5.8	---	X	---	---	DOP-26

^aBased upon nominal 40.64 mm diam, except MHFT-62 through -66, which are based upon Mound Facility gaging.

^bParenthesis indicate penetration confirmed metallographically.

TABLE II. AES Analysis of Multi-Hundred Watt Iridium

Specimen ^a	Location	Th ₆₅ /Ir ₂₂₉	P ₁₂₀ /Ir ₂₂₉	C ₂₇₀ /Ir ₂₂₉	O ₅₁₀ /Ir ₂₂₉	Th ₆₅ /Ir ₅₄	Other
MHFT-71-A1	Inside edge	0.26	---	0.59	0.45	0.04	---
	Center	0.62	---	0.57	0.70	0.09	---
	Outside edge	0.62	0.18	0.67	0.85	0.10	---
MHFT-71-A2	Inside edge	0.04	---	0.99	0.38	Trace	---
	Center	0.36	---	1.20	0.99	0.06	---
	Outside edge	0.34	---	1.69	1.29	0.07	0.51 ^b Trace ^c
MHFT-71-B1	Inside edge	0.13	---	0.58	0.40	0.02	---
	Center	0.26	---	0.59	0.65	0.04	---
	Outside edge	0.13	---	0.62	0.68	0.04	---
MHFT-71-B2	Inside edge	0.35	---	0.76	0.85	0.17	0.42 ^b
	Center	0.03	---	1.13	0.90	0.004	0.60 ^b
	Outside edge	Trace	---	1.33	0.84	Trace	0.55 ^b

^aTreatment of specimen: 9215 h at 1210°C GIS.

^bSi₁₉₂/Ir₂₂₉.

^cSi₁₅₀/Ir₂₂₉.

TABLE III. Summary of AES Results of the Iridium Clad of MHFT-71

Location	Th ₆₅ /Ir ₂₂₉	P ₁₂₀ /Ir ₂₂₉	Si ₁₅₀ /Ir
Inside edge	0.20	---	---
Center	0.32	---	0.21
Outside edge	0.27	0.05	0.27
Average	0.26	0.02	0.16

B. Environmental Exposure

A moderate sized fragment of the plutonia sphere from the assembly FSA MHFT-12 (SPO-32), exposed in the Los Alamos Group CMB-1 environmental chamber for 7 yr, was examined. As received, the fragment was coated with a loosely agglomerated coating of soil, shown in Fig. 7a. The soil was removed by scraping until only a thin, relatively adherent coating remained. A photograph of the fragment after it was broken in two is shown in Fig. 7b.

Ceramographic examination of the sample showed the plutonia fragment to be of uniform density with the angular, elongated pores illustrated in the photomicrographs of Fig. 8. Because of the irregular surface

configuration of the sample, it is difficult to determine with certainty whether or not surface corrosion or spallation of plutonia particles has occurred. However, no specific evidence of such degradation was detected. Photomicrographs at four locations along the surface are shown in Fig. 9. Many of the near-surface pores are filled with nonmetallic deposits. Analysis indicated the presence of potassium, calcium, silicon, aluminum, iron, and sodium in this material. These elements are common constituents of soil minerals, and their presence in the pores suggests a mechanism of dissolution by the simulated rain and reprecipitation under the influence of the elevated temperature of the plutonia during the dry cycles.

Unfortunately, the relatively adherent agglomerated soil coating did not survive the polishing process, and no estimate of its thickness could be made.

IV. HELIUM RELEASE (D. Peterson, J. Starzynski)

The measured helium release rates from 18 samples of ²³⁸PuO₂ were analyzed this month. The samples were from GPHS fuel pellet GP-19, which previously had been subjected to a simulated reentry thermal pulse. In this study, samples were exposed in a vacuum to a thermal pulse in which the temperature was increased

from 450° to 1510°C in 280 s. The helium intensity was monitored continuously throughout this period. The temperature was then increased to 1600°C and maintained there until the helium signal dropped to background.

The release rates were modeled using Fick's laws of simple diffusion through the bulk. This treatment gave an effective diffusion constant, D' (equal to D/a^2 , where a is the radius of the releasing unit). D' is related to the activation energy for diffusion, E_a , by the relationship,

$$D' = D'_0 e^{-E_a/RT}$$

where D'_0 = effective constant of diffusion, and R = gas constant.

A plot of $\log D'$ vs reciprocal temperature for the average of the 18 samples is shown in Fig. 10. (The ranges in the data are indicated by the bars.) Also plotted are the results of Mulford and Mueller² on microspheres and pellets and the results on microspheres by Angelini, McHenry, Scott, and Prados.³ The comparison data were obtained by monitoring the helium release from

isothermally heated samples. It is encouraging to note that the isothermal results on pellets and microspheres agree quite well with the high-temperature D' values obtained from the thermal ramps in this study. The low-temperature data appear to show more dependence upon particle size.

The data from each sample exhibited the two-slope character shown in the figure. The results of linear least squares analyses of the two regions of each sample are summarized in Table IV. The data analysis assumed that the releasing volumes were all the same size. A multiple-size model is being developed.

Samples from the outer and center regions of GPHS pellet GP-19 were subjected to the thermal pulse without the final 1600°C treatment. The heat-treated samples were prepared ceramographically and compared to a control sample that had not been exposed to the 280-s pulse. Figure 11 shows the microstructures of the samples following the pulse. All three samples show evidence of helium bubble formation, presumably from the previous reentry ramp. It is evident that the center sample shows less helium bubble damage, a smaller

TABLE IV. Activation Energies and Effective Constants of Diffusion for Helium Release from ²³⁸PuO₂ Exposed to a Thermal Ramp (450°C-1510°C/280 s)

Run No.	Mass (g)	Age (yr)	Total Helium (%)	Low Temperature		High Temperature	
				E_a (kcal)	$\log D'_0$ (s ⁻¹)	E_a (kcal)	$\log D'_0$ (s ⁻¹)
17	0.200	0.520	100.0	30.1±1.6	-3.834±0.368	67.0±2.8	3.574±0.448
18	0.169	0.529	87.4	17.7±0.4	-5.398±0.094	63.8±2.4	3.471±0.410
19	0.255	0.532	90.2	17.7±1.0	-5.341±0.244	59.8±2.7	2.697±0.454
20	0.236	0.532	89.4	21.5±2.4	-6.195±0.602	62.5±4.4	2.712±0.651
21	0.231	0.534	88.8	21.6±2.2	-4.499±0.533	62.4±2.4	3.713±0.409
23	0.118	0.537	90.8	27.0±1.4	-4.541±0.310	67.6±1.5	3.906±0.230
24	0.116	0.551	94.0	29.4±1.4	-3.355±0.295	64.6±2.1	3.463±0.308
25	0.116	0.553	90.9	23.5±1.4	-4.494±0.320	62.5±2.0	3.237±0.326
26	0.368	0.553	91.6	23.9±1.7	-5.392±0.375	62.8±1.8	2.752±0.249
28	0.387	0.567	89.0	22.3±0.4	-5.184±0.102	65.0±4.0	3.304±0.527
29	0.406	0.570	93.2	25.2±1.1	-4.784±0.257	62.1±2.4	3.058±0.334
30	0.450	0.570	94.2	21.6±0.5	-5.736±0.117	59.9±2.1	2.394±0.291
31	0.476	0.597	95.4	24.5±0.3	-5.896±0.080	61.9±2.7	2.434±0.374
32	0.599	0.605	96.1	24.7±2.0	-5.257±0.472	68.9±3.4	3.610±0.502
33	0.492	0.608	82.7	34.2±1.3	-3.448±0.309	61.6±3.9	2.780±0.597
36	0.156	0.652	90.6	---	---	67.4±2.0	4.080±0.324
38	0.253	0.663	86.8	27.1±5.4	-4.596±1.22	69.5±1.5	3.556±0.208
40	0.744	0.707	83.1	25.0±1.8	-6.942±0.463	61.5±2.2	2.925±0.293
Average =				24.5±1.5	-4.994±0.362	63.9±2.6	3.209±0.385

average grain size, and a higher porosity than the outer edge samples.

A sample of GPHS fuel pellet GP-19 was analyzed by emission spectrometry. This fuel had been previously subjected to a simulated prelaunch heat treatment of 1135°C for 5 days followed by a thermal pulse. The results of the analysis are given in Table V. The calcium

content is significantly higher than the 300 ppm specified for the shards used to make the pellets.

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3. P. Angelini, R. E. McHenry, J. L. Scott, W. S. Ernest, and J. W. Prados, "Helium Release from $^{238}\text{PuO}_2$ Microspheres," Oak Ridge National Laboratory report ORNL-4507 (March 1970).

TABLE V. Results of Spectrochemical Analysis of GPHS Fuel Pellet (GP-19)

Li	<1	Be	<1	B	1	Na	5
Mg	150	Al	<5	Si	150	K	<5
Ca	900	Ti	150	V	<3	Cr	20
Mn	2	Fe	150	Co	<3	Ni	5
Cu	<1	Zn	20	Rb	<10	Sr	<5
Y	<25	Zr	<100	Nb	<10	Mo	<3
Ag	7	Cd	<10	Sn	<5	Ba	<2
La	<25	Hf	<25	Ta	100	W	<10
Re	<25	Pb	<5	Bi	<1

(a)

(b)

(c)

(d)

Fig. 1. The weld-overlap regions of the capsules used in the DIT-2 impact were uniform: (a) SR-105, 8X; (b) SR-106, 5X; (c) SR-107, 5X; and (d) SR-110, 5X.

Fig. 2. The thermocouple temperature histories of the DIT-2 impact heating period.

Fig. 3. The thermal histories of four LWRHUs compared to the desired history. (a) RU-9-2; (b) RU-9-3; (c) RU-9-4; and (d) RU-9-6.

Fig. 4. The tantalum impact can of MHFT-71 was unsymmetrical after the impact.

Fig. 5. The iridium clad on MHFT-71 was dull gray when it was removed from the impact shell. (a) Impact face (2X); (b) back side (1X); and (c) profile (1X).

Fig. 6. Typical microstructures of the iridium clad of MHFT-71, 50X.

(a)

(b)

Fig. 7. Photographs of the fragment of the plutonia sphere from MHFT-12 (SPO-32) after 7-yr exposure in the environmental chamber. (a) With loosely agglomerated crust, ~2X. (b) Scraped and fractured, 2X.

Fig. 8. Microstructure of the plutonia fragment from MHFT-12.
100X as polished.

(a)

(b)

(c)

(d)

Fig. 9. Micrographs of the surface regions of the fragment of plutonia sphere (SPO-32) after 7-yr exposure in environmental chamber, all 250X.

Fig. 10. Plot of log of the effective diffusion constant versus reciprocal temperature for helium release from plutonia.

(a)

(b)

(c)

Fig. 11. Microstructures of plutonia samples removed from GPHS pellet GP-19 and exposed to 450-1510°C/280-s pulse. (a) Outer edge; (b) center; and (c) outer edge, control. 500X, as etched.

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