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H-12-83
LA-9715-PR
Progress Report

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I-8753

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Space Nuclear-Safety Program

October 1982

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This work was supported by the US Department of Energy, Office of Special Nuclear Projects.

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LA-9715-PR
Progress Report.

UC-33¹
Issued: March 1983 ✓

LA--9715-PR

DE83 009644

Space Nuclear-Safety Program

October 1982

Compiled by
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SPACE NUCLEAR SAFETY PROGRAM

October 1982

Compiled by
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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 Special Nuclear 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.

I. GENERAL-PURPOSE HEAT SOURCE

A. DIT-3 Impact (F. W. Schonfeld)

The post-mortem evaluation of the General-Purpose Heat Source (GPHS) module used in the third design iteration test (DIT-3) was completed. The purpose of this test was to determine if weld cracks of sizes 8.0, 9.0, 10.4, and 11.5 (arbitrary units) would affect the fuel retention capability of the GPHS iridium clad during a 30° impact. None of the four fueled clads failed, and there was no evidence that any weld crack was enlarged by the impact.

1. Iridium Chemistry. A sample of the side wall of each half of the four capsules used in the DIT-3 module was analyzed spectrochemically, and a sample of each cup and of the weld between them was analyzed by Auger Electron Spectroscopy (AES).

The elements detected on the spectrochemical surveys are listed in Table I. The differences among the samples are not large enough to have caused differing mechanical responses during the impact. Further, the impurity levels in the DIT-3 capsules are not significantly different from those in DIT-1 or DIT-2.

The elements on the freshly fractured surfaces detected by AES are listed in Table II. Again, the elements and their amounts found on the fracture surfaces do not differ greatly for the 12 samples examined, nor are they significantly different from those detected on the examined surfaces of DIT-1 and DIT-2. Neither the small differences in impurity contents nor the occasionally detected oxygen, carbon, sulfur, and chlorine or boron (number 180) affected the impact responses of the four clads.

2. Iridium Hardness. The results of hardness tests on samples of each cup and weld of the four capsules used in DIT-3 are listed in Table III. No trends in hardness across the capsule walls were detected. The hardness of the recrystallized DOP-26 iridium alloy used in the GPHS capsules is usually in the range $275 \pm \text{DPHN}$. Most of the samples from DIT-3 exceeded that range, suggesting that strain hardening accompanied the impact deformations.

3. Fuel Chemistry. The results of the spectrochemical and phosphorus analyses of the fuel pellets used in the DIT-3 module are listed in Table IV. The levels of aluminum and tantalum in pellet SRP-127 and

TABLE I. Results of Spectrochemical Analyses of DIT-3 DOP-26 Iridium-Alloy Cups (2000 ppm Tungsten)

Elements ^a	SRP-127		SRP-140		SRP-251		SRP-317	
	V Cup ^b	WS Cup ^c	V Cup	WS Cup	V Cup	WS Cup	V Cup	WS Cup
Al	50	80	50	50	50	50	30	40
Ca	3	---	---	---	---	---	---	---
Cr	10	10	---	---	40	50	---	---
Cu	20	30	25	35	8	10	30	25
Fe	100	100	80	100	200	200	100	100
Mg	20	6	10	---	---	---	7	3
Ni	30	50	40	50	50	50	40	50
Pt	---	30	---	---	---	---	---	---
Si	20	15	10	---	10	10	20	10
W	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴	10 ³ -10 ⁴

^aOnly the elements present in amounts exceeding their limits of detection are listed.

^bVent cup.

^cWeld-shield cup.

tantalum in SRP-140 are higher than usual, but we could discern no effect caused by these departures from the norm.

4. Fuel Breakup. The particle size analysis of fuel pellet SRP-317 from the DIT-3 impact is compared with the analyses of pellets SRP-48 from DIT-1 and SRP-110 from DIT-2 (Table V). There are no significant differences among them.

5. Conclusions. The DIT-3 impact was intended to determine if the size of closure-weld cracks would affect the fuel containment capability of the GPHS cladding at a 30° impact angle. At that angle the insult to the fueled clads was not sufficient to load the weld areas to the failure point; therefore, no effect on failure was seen. We saw no evidence that the impact had enhanced any crack.

Comparing the DIT-3 results with the DIT-1 and DIT-2 results provides strong evidence that the flat-on orientation is the most severe impact orientation within the assumed 30° wobble.

B. Early Compatibility Test (D. Pavone)

This particle-module early compatibility test (ECT) assembly was aged with the clad temperature at 1310°C in vacuum for 6 months and impacted at 930°C and

59.3 m/s. The fueled clads were identified as HF2 and HF5. Briefly, HF2 sustained a major failure on the backside that extended onto the vent-end face and other cracks near the vent assembly. A less severe failure occurred on the vent-end face of HF5. Metallographic examination of a transverse cross section of the HF2 vent cup (LR234-1) revealed a highly variable microstructure with 1-2 grains across the clad thickness near the failures; in other regions a duplex microstructure had large grains on the interior 50% of the wall and small grains on the exterior (the microstructure expected after the 6-month aging) and an inverse large-grain/small-grain relationship at some locations. The shield-cup microstructure (LR231-1) was uniform around the circumference, exhibiting the expected duplex grain size across the clad.

Examination of the interior surfaces of the weld beads on the impact faces revealed cracks in both capsules. Photomacrographs (Fig. 1) show the interior surfaces of the weld beads of capsules HF2 and HF5. The HF2 weld overlap was located at the reentrant fold of the capsule. We observed two minor cracks at this location within the weld bead, and additional cracks (a few extending into the parent metal) occurred nearby. The HF5 weld bead exhibited a higher density of weld-bead cracks, as well as cracks in the parent metal. The weld overlap of this capsule was located at the edge of the impact face about 90° from the reentrant fold.

TABLE II. Post Impact Auger Intensity Ratios

Specimen	Site	Location	Th _{6s} /Ir ₅₄	Th _{6s} /Ir ₂₂₉	C ₂₇₀ /Ir ₂₂₉	O ₅₁₀ /Ir ₂₂₉	S ₁₅₀ /Ir ₂₂₉	Cl or B ₁₈₀ /Ir ₂₂₉
SRP 127 A	Vent cup wall	Inside edge	0.045	0.476				
		Center	0.068	0.645	0.488	0.714		
		Outside edge	0.048	0.472	0.427	0.697		
SRP 127 B	Weld shield cup wall	Inside edge	0.063	0.572				
		Center	0.072	0.810				
		Outside edge	0.052	0.590				
SRP 127 C	Weld	Inside edge	0.061	0.5895				
		Center	0.083	0.804				
		Outside edge	0.057	0.667				
SRP 140 A	Vent cup wall	Inside edge	0.030	0.290				
		Center	0.045	0.432				
		Outside edge	0.041	0.354				
SRP 140 B	Weld shield cup wall	Inside edge	0.084	0.835				
		Center	0.067	0.627				
		Outside edge	0.051	0.468				0.053
SRP 140 C	Weld	Inside edge	0.058	0.550		0.813		0.099
			0.052	0.495		0.667		0.054(Cl)
		Center	0.023	0.220	0.132	0.769		
		Outside edge	0.051	0.452	0.280	1.032		
		Inside edge	0.075	0.744				
SRP 251 A	Vent cup wall	Center	0.093	1.000				
		Outside edge	0.076	0.727				0.068(Cl)
		Inside edge	0.039	0.416				
SRP 251 B	Weld shield cup wall	Center	0.076	0.763				
		Outside edge	0.060	0.564				0.090
		Inside edge	0.045	0.463				
SRP 251 C	Weld	Center	0.061	0.684				
		Outside edge	0.051	0.535				
		Inside edge	0.066	0.644				
SRP 317 A	Vent cup wall	Center	0.096	0.876				
		Outside edge	0.068	0.625		0.417		
		Inside edge	0.057	0.539		0.382		
SRP 317 B	Weld shield cup wall	Center	0.072	0.773				
		Outside edge	0.058	0.365		0.544		
		Inside edge	0.046	0.435				
SRP 317 C	Weld	Center	0.063	0.651				
		Outside edge	0.080	0.824				

Table III Hardness of the Iridium Alloy Capsules Used in the DIT 3 Impact, DPHN (200 g)

Specimen			DPHN at 95% C.L. ^a	Range
SRP 127	A	Vent cup	291 ± 11	257 – 317
	B	Weld shield cup	291 ± 9	258 – 308
	C	Weld	302 ± 16	275 – 369
SRP 140	A	Vent cup	250 ± 7	237 – 273
	B	Weld shield cup	307 ± 27	259 – 376
	C	Weld	273 ± 7	262 – 291
SRP 251	A	Vent cup	280 ± 10	256 – 306
	B	Weld shield cup	285 ± 7	267 – 303
	C	Weld	283 ± 12	262 – 318
SRP 317	A	Vent cup	293 ± 14	266 – 342
	B	Weld shield cup	234 ± 9	219 – 253
	C	Weld	270 ± 11	244 – 297

^aConfidence level

TABLE IV. Post-Impact Spectrochemical Survey of DIT-3 Fuels

Elements ^a	Pellet No.			
	SRP-127	SRP-140	SRP-251	SRP-317
Ag	4	5	15	20
Al	260	35	45	35
B	1	<1	<1	<1
Ba	4	<2	<2	2
Ca	80	80	80	80
Cr	5	20	20	<5
Fe	85	140	135	65
Mg	1	<1	1	<1
Mn	1	1	1	<1
Na	20	3	4	3
Pb	5	5	<5	8
Si	45	15	55	20
Ta	1000	500	100	100
Ti	50	50	50	25
P ^b	<20	<20	<20	<20
P ^c	15	10	13	20

^aElements are listed only if they exceed detectability limits in at least one fuel pellet.^bGamma-scan result.^cWet-chemistry result.

TABLE V. Size Distributions of Impacted Fuel

Particle Size (μm)	Weight Fraction		
	DIT-1 (SRP-48)	DIT-2 (SRP-110)	DIT-3 (SRP-317)
+6000	0.3702	0.4620	0.4671
+2000	0.3713	0.3463	0.3371
+841	0.1596	0.1269	0.1236
+420	0.0426	0.0304	0.0483
+177	0.0173	0.0125	0.0035
+125	0.0036	0.0027	0.0044
+74	0.0062	0.0042	0.0001
+44	0.0073	0.0031	0.0049
+30	0.0095	0.0043	0.0045
+20	0.0033	0.0024	0.0034
+10	0.0069	0.0044	0.0023
-10	0.0022	0.0009	0.0010

(a)

(b)

Fig. 1. Photographs of the interior of the weld bead in capsules used for the ECT. (a) HF2 and (b) HF5 in which the vertical band at the left is a piece of the weld shield. Both $\sim 8X$.

Examination of transverse metallographic cross sections of the HF5 cups revealed variations in the microstructure of the vent cup (LR234-3) similar to those observed in HF2. The microstructure in the shield cup was generally uniform, although we observed isolated large grains. Figure 2 illustrates the range of microstructures observed in the HF5 iridium vent cup. The clad failure (Fig. 2a) demonstrates the brittle fracture mode and the extremely large grain size at this location. Figure 3 shows the microstructures observed in the shield cup. Except for isolated instances of large grains, the microstructure is what would be expected after the aging treatment.

Results of AES analysis of the grain boundary chemistry of samples from the iridium clads are listed in Table VI. Absorbed current images indicated that all samples were taken at the normal duplex large-grain/small-grain microstructure locations. The results indicate a general depletion of thorium at the grain boundaries, particularly at the inside surface; however, in one HF5 sample the thorium concentration was uniform across the thickness and was near the expected level. Carbon and oxygen were the principal impurity elements detected, occurring in four of the seven samples analyzed. We observed a boron or chlorine peak in two samples and silicon at one location. Neither phosphorus nor sulfur, elements which may be factors in abnormal grain growth, was detected.

Table VII gives the results of the spectrographic analyses of the iridium cups used in the ECT impact. None of the impurity elements detected is present in abnormally high concentrations.

A sample of each plutonia pellet was analyzed for cationic impurities by emission spectrographic analysis and for phosphorus by a chemical procedure. Table VIII lists those elements present in amounts exceeding the detection limit and compares the corresponding Savannah River analyses of samples of the shards used to make the pellets. It is interesting to note that in most cases the impurity levels detected in the pretest analyses (Savannah River) were higher than those detected in the post-test analyses (Los Alamos).

Sieve analyses of the fuel remaining in the clads are listed in Table IX. They demonstrate that HF5 was less severely broken up than HF2, with 85% of and 55% of the pellet weights above 2000 μm , respectively.

Analysis of the recovered graphite impact shell for total plutonium indicated that 0.318 g of plutonium had been expelled from the capsules.

The analytical data collected thus far does not point to a specific cause for the abnormal and variable microstructures observed in the vent cups of both fueled clads in this test. In reviewing data collected during the Galileo multihundred watt (MHW) test series, we noted that the iridium microstructure at the site of a hoop failure, located near one of the vent assemblies of MHW MHFT-73, consisted of large grains but that, at the site of the fuel push-through failure, the iridium microstructure was the duplex grain structure. To investigate the possibility that the growth of the large grains was related to proximity to the vent assembly, we examined a metallographic cross section of the MHFT-73 clad. Our examination included a portion of one of the vent assemblies and extended to the equatorial weld. The microstructures observed in this sample are illustrated in Fig. 4. Figures 4a and 4b show the microstructure at the site of the failures. The Fig. 4c photomicrograph, located within the vent assembly, shows an area of relatively small grains adjacent to an extremely large grain. The microstructures at about 45° from the vent and near the weld are shown in Figs. 4d and 4e, respectively. The latter microstructures, in contrast to those at the failure sites and within the vent assembly, consist of moderately large grains (~4 grains/thickness) and are uniform across the thickness of the hemishell. This observation indicates that variations in microstructure of the DOP-26 clad are not unique to the vent cups of the ECT and may have been more widespread than previously thought.

C. Phosphorus Effects Experiment (D. Pavone)

On November 1, 1982, the accumulated exposure time for the fueled assemblies was 3265 h, and the exposure time for the control samples was 2249 h.

The AES results for archive-ring control sample P714-5 were

$$\frac{\text{Th}_{65}}{\text{Ir}_{229}} \text{ intensity ratio} = 0.67,$$

$$\frac{\text{O}_{510}}{\text{Ir}_{229}} \text{ intensity ratio} = 0.46, \text{ and}$$

$$\frac{\text{Th}_{65}}{\text{Ir}_{54}} \text{ intensity ratio} = 0.07.$$

(a)

(d)

(b)

(e)

(c)

Fig. 2. Microstructures observed in the transverse cross section of the HF5 vent cup. All 50X.

(a)

(d)

(b)

(e)

(c)

Fig. 3. Microstructures observed in the HF5 shield cup. (a), (b), and (c) are typical; (d) and (e) represent isolated large-grain observations. All 50X.

TABLE VI AES Results for the ECT Iridium Cups

Specimen	Location	Th ₆₅ /Ir ₂₂₉	P ₁₂₀ /Ir ₂₂₉	C ₂₇₀ /Ir ₂₂₉	O ₅₁₀ /Ir ₂₂₉	Si ₉₂ /Ir ₂₂₉	S ₁₅₀ /Ir ₂₂₉	B or Cl ₁₈₀ /Ir ₂₂₉	Th ₆₅ /Ir ₅₄
HF2	Int edge	0 18		1 18	0 85				0 03
Vent cup 1	Center	0 36		0 89	1 04				0 04
	Ext edge	0 43		1 64	1 13				0 10
	Average	0 32		1 24	1 01				0 06
HF2	Int edge	0 20							0 02
Vent cup 2	Center	0 67							0 06
	Ext edge	0 38						0 08	0 04
	Average	0 42						0 03	0 04
HF2	Int edge	0 31		0 42	0 92			0 60	0 03
Shield cup 1	Center	0 29			0 87			0 12	0 03
	Ext edge	0 34		0 49	0 83			0 16	0 05
	Average	0 31		0 30	0 87			0 29	0 04
HF5	Int edge	0 19							0 02
Vent cup 1	Center	0 85							0 09
	Ext edge	0 60							0 07
	Average	0 55							0 08
HF5	Int edge	0 37							0 06
Vent cup 2	Center	0 62							0 05
	Ext edge	0 68				0 50			0 07
	Average	0 57				0 17			0 06
HF5	Int edge	0 09							0 01
Shield cup 1	Center	0 89							0 08
	Ext edge	0 71			0 88				0 08
	Average	0 56			0 29				0 06
HF5	Int edge	0 41		0 53	0 56				0 04
Shield cup 2	Center	0 37		0 51	0 73				0 04
	Ext edge	0 51		0 38					0 05
	Average	0 43		0 47	0 43				0 04

TABLE VII. Emission Spectrographic Analysis of Iridium Clads Used in ECT

Element ^a	Impurity Content (ppm)			
	HF2		HF5	
	Vent Cup	Shield Cup	Vent Cup	Shield Cup
Chromium	50	40	40	40
Magnesium	5	5	5	nd
Nickel	100	100	100	100
Aluminum	70	70	60	80
Copper	10	15	10	20
Calcium	3	nd ^b	3	nd
Iron	250	250	300	300
Silicon	50	30	50	50
Platinum	30	30	50	50
Rhodium	30	30	30	50

^aElements not listed were below detection limits.^bBelow detection limit.

TABLE VIII. Chemical Analyses of the Fuel Used in ECT (The numbers in parentheses are the analyses determined at Savannah River.)

Element	Impurity Content (ppm)	
	Pellet HF2	Pellet HF5
Silver	5	5
Silicon	45 (170)	8 (78)
Aluminum	25 (268)	160 (248)
Calcium	200 (75)	140 (50)
Copper	60 (15)	1 (5)
Lithium	2	1
Iron	40 (441)	160 (678)
Magnesium	1 (20)	1 (10)
Manganese	1 (15)	1 (100)
Zinc	15 (20)	5 (2)
Titanium	5	10
Chromium	20 (150)	85 (175)
Tungsten	nd ^b	10
Tantalum	nd	200
Phosphorus ^a	8	6

^aNot spectrographic.

^bBelow detection limit.

TABLE IX. Size Distribution of Fuel Remaining in the ECT Clads

Particle Size (μm)	Accumulated Weight Fraction	
	Pellet HF2	Pellet HF5
+6000	0.2081	0.5791
+2000	0.5469	0.8504
+ 841	0.7963	0.9071
+ 420	0.8940	0.9412
+ 177	0.9428	0.9605
+ 125	0.9519	0.9647
- 125	1.0000	1.0000

The Th/Ir intensity ratios are normal for DOP-26 iridium.

D. Explosion Tests

The Los Alamos GPHS overpressure experiment was completed as part of the Defense Nuclear Agency's pre-

DIRECT-COURSE event, and preparations were made for the Sandia National Laboratories' shock-tube experiments.

1. Pre-DIRECT-COURSE. (R. E. Tate and R. W. Zocher) The pre-DIRECT-COURSE event was a Defense Nuclear Agency explosion completed on October 7, 1982, at the White Sands Missile Range. In this test a 40 000-lb (TNT equivalent) explosive charge of ammonium nitrate/fuel oil (ANFO) contained in a 3.66-m-diam (12-ft) sphere was detonated atop a 15.24-m (50-ft) tower. The test provided an explosion source for several experiments and is a precursor to the DIRECT-COURSE test in 1983 in which 0.55×10^6 kg (1.2×10^6 lb) of ANFO will be detonated.

The Los Alamos sample consisted of a stack of three simulated GPHS modules. The center module was made of an aeroshell, two impact shells, and four lead-alloy mass simulants. The modules on either side were machined to the correct outer shape from solid graphite. The internal components of the prime module were painted fluorescent orange, and the external components and the bulk-graphite simulants were painted fluorescent green. We hoped that the colorful paint would make the

(a)

(d)

(b)

(e)

(c)

Fig. 4. Microstructures observed in MHW MHFT-73. (a) and (b) failure sites, (c) near pole, (d) $\sim 45^\circ$ from the vents, and (e) near equator. All 50X.

objects easier to detect in photographs during the explosion and that it would make the modules or pieces of them more visible during the post-test search of the test site. The module stack was mounted on a plastic pipe and located 3.96 m (13 ft) from the surface of the glass-fiber-reinforced plastic sphere used to contain the ANFO. At this position, the pressure was supposed to be 12.8 MPa (2000 psi).

Approximately 2 h after the detonation we were able to enter the test site and search for our samples. During the 2-day search, we found 16 sample pieces: 10 pieces of the bulk graphite used for the outer blocks and 6 pieces of the carbon composite used for the central module. One of the latter was from the impact shell; the other five were from the aeroshell.

Figure 5 is a map of the site showing the points at which the fragments were located. The test area was searched visually to a range of 550 m (1800 ft). On the second day, a metal detector was also used in an unsuccessful search for the lead-alloy fueled clad simulants.

When recovered, the graphite fragments were essentially devoid of the fluorescent paint, which had vaporized in the explosion fireball. Figure 6 shows the two largest composite fragments recovered. They are mating pieces of one side of the aeroshell. The remains of the internal threads can be seen in the opposite ends of the two pieces. The ciné photographs revealed that the test object's location was within the fireball, so the lack of paint is not astonishing.

The experiment revealed that the 12.8-MPa (2000-psi) force was sufficient to destroy a GPHS module that had no additional protection. There was a 10% chance that a

fragment of the plastic shell around the ANFO hit the test module. The size of the fireball and the lack of detail in the ciné films prevent a definitive evaluation of this possibility.

2. Shock Tube Tests. (R. W. Zocher) The prototype furnace for use in the overpressure tests to be conducted at the Sandia National Laboratories' shock tube facility was completed. Figure 7 shows the furnace and test object. The stack of modules will be suspended on the trapeze, which is inverted in the figure and surrounded by the furnace in the end of the shock tube. The module will be heated to 1091°C and stabilized at that temperature. Immediately before detonation of the explosive in the breach of the shock tube, the furnace will be dropped into a protected recess in the bottom of the tube, leaving the test module suspended in the center, exposed to the shock wave.

II. Helium Release (D. Peterson)

Results from analyses of more than 60 experiments performed last year on the helium release behavior of PuO_2 were correlated this month. The results and theoretical models have been incorporated into a report describing the current status of our research in this area.

We completed a plan for measuring the helium release from a 5-yr-old pellet. The thermal pulse will be controlled by a minicomputer, and the release rate and temperature will be measured simultaneously in an automated mode.

Fig. 5. Sixteen fragments of the three module stack were recovered after the explosion FWPF is Fineweave Pierced Fabric 3 D carbon/carbon composite, a product of AVCO Systems Division, 201 Lowell St., Wilmington, MA 01887

GPHS
PRE-DIRECT-COURSE TEST
AEROSHELL FRAGMENTS

F-2

F-4

174 m 310 m
RADIAL DISTANCE

Fig. 6. The two largest fragments recovered came from the side wall of the aeroshell.

Fig. 7. The three-module stack will be heated in the shock tube.