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Progress Report

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Space Nuclear Safety and Fuels Program

August 1981

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Compiled by

S. E. Bronisz

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SPACE NUCLEAR SAFETY AND FUELS PROGRAM

AUGUST 1981

Compiled by

S. E. Bronisz

ABSTRACT

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

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

I. GENERAL-PURPOSE HEAT SOURCE

A. Impact Test—Design Iteration Test-1.5 (R. W. Zocher)

The current schedule for the Design Iteration Test (DIT)-1.5 impact test is shown in Fig. 1. The critical item continues to be receipt of the fueled clads, but all other components are on schedule. Figure 2 shows the partial aeroshell for this test before outgassing.

The carbon-bonded, carbon-fiber (CBCF) sleeve 7787-28-5839 that was selected for use in the DIT-1.5 impact cracked during the 36-h, 1500°C vacuum outgassing. This sleeve was designated by Oak Ridge National Laboratory (ORNL), its manufacturer, for test use because it was 0.025 mm longer than specified; it had no other deviant characteristics. The crack, shown in Fig. 3, was longitudinal and extended along 40% of the sleeve's length. Although changes in weight and dimension were observed in the other CBCF sleeves outgassed at Los Alamos, none before have cracked after outgassing.

B. Postmortem Examination DIT-1 (F. W. Schonfeld)

1. Vent Characterization. The vents from each of the four fueled clads used in the DIT-1 impact were sectioned and examined microscopically to determine if the pretest-aging treatment of 100 h at 1300°C had caused material to be deposited in the vent structures and to define their responses to the impact. The interior surfaces of all four capsules were blackened by thin films of PuO_2 , but no fuel or fuel impurities were detected in any of the four vent cross sections. Additionally, no mechanical defects were found, and all the vents appear to have survived the test without significant harm. Figure 4 shows the microstructure of the vent from IRG-102, which is typical of the four vents. As Fig. 4 suggests, the vent areas were deformed little during the test; apparently they were well protected by being held firmly between the fuel pellets and the FWPF* graphite partitions.

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

2. Iridium Alloy Characterization. The capsule components used in the DIT-1 test were given two heat treatments, a grain-size anneal before welding and a weld anneal after assembly. The purpose of the grain-size anneal of 20 h at 1500°C at Mound Facility was to produce the same grain size in the test capsules as flight capsules would have after 6 months of operation. The combination of time and temperature was specified by ORNL and was supposed to produce a grain size of 13 to 14 across the 0.64-mm thickness of the iridium.

The weld anneal of 100 h at 1300°C also was specified by ORNL and was performed at Los Alamos on impact capsules IRG-101 through IRG-104 after welding (example-weld capsules IRG-105 and IRG-106 were not weld annealed). The purpose of the weld anneal was to increase the ductility of the welded metal by exposing it to the normal acceptance-test conditions for the production of General-Purpose Heat Source (GPHS) iridium capsules. Grain size was not affected.

Grain sizes of the six DIT-1 capsules, two example-weld capsules and four impacted capsules, are listed in Table I. All 12 cups making up the 6 capsules had grains significantly smaller than predicted, but we cannot explain this discrepancy. The smaller variations in grain size from cup to cup are to be expected because the iridium alloy is made in small batches. If the grain size had been 13 to 14, as predicted by ORNL, the iridium would have been less ductile and the cracks would have been more severe than those in the four DIT-1 capsules.

Representative microstructures of the 12 cups are shown in Figs. 5 to 10. Each cup exhibited the normal structural features, and the iridium appeared to be of good quality and reasonable reproducibility.

The hardness of each capsule was measured with a standard diamond pyramidal indenter under a 200-g load. The measurements were made in both undeformed and deformed portions of the cups and on the welds that joined the cups (see Table II).

Results indicate that because the 930°C impact temperature was below the hot working temperature significant strain hardening resulted from the impact. Some stress relief probably occurred during the 18 h between impact and recovery of the DIT-1 module; therefore, the immediate strain hardening was reduced to the values observed here. The hardness results also indicate that example-weld capsules IRG-105 and IRG-106 were not as hard as the impacted capsules.

Their treatment differed from that of the impact capsules in two ways: the weld anneal and the impact. Of the two, the difference in impact is more likely to cause hardness variation between the impacted and unimpacted capsules; that is, the "undeformed" regions of the impacted capsules are really regions of low deformation.

Samples of each cup of the six capsules were submitted for spectrographic analysis, and four samples of each capsule (two from cup walls, one perpendicular to and crossing the weld centerline and one parallel to and including the weld centerline) were prepared for Auger analysis. These results, summarized in Tables III and IV, suggest that the six capsules had similar chemistries, with all variations well within the range one might reasonably expect from small-lot preparations.

3. Fuel Characterization. Chemical analysis of the four fuel pellets impacted in DIT-1 is given in Table V. The phosphorous contents are reasonably low; the wet-chemistry results compare well with the results of the radioanalysis done on the whole pellets. Three pellets, those in capsules IRG-102, IRG-103, and IRG-104, had levels of boron, magnesium, and calcium impurities that exceeded the limits allowed in sintered shards. IRG-102 was high in silicon.

The particle-size distribution of the fuel from capsule IRG-102 was determined (see Table VI). The proportion of fines is not significantly different from what we have seen before.

II. LIGHT-WEIGHT RADIOISOTOPE HEATER UNITS

A. Test Samples (R. Tate)

Eighteen Light-Weight Radioisotope Heater Units (LWRHU) delivered for safety tests include eight fueled units and ten units containing UO_2 pellets. Nine units, six fueled with PuO_2 and three with UO_2 , are made with flight-quality graphite assemblies. The other nine units were assembled with pyrolytic graphite insulator sleeves that have imperfect standoff rings. The LWRHUs made with flight-quality graphite will be used in safety tests requiring dynamic thermal environments, and those with the imperfect graphite will be used in tests not requiring such environments.

B. Reentry Tests (C. Frantz)

An electron-beam furnace for LWRHU reentry tests was designed and fabricated. It performed satisfactorily in the proof tests and now is ready for the LWRHU tests.

C. Explosion Tests (C. M. Seabourn)

This month a final attempt was made to increase the impulse length beyond 18.6 kPa·s (2.7 psi·s), the longest pulse yet attained. In this attempt, the sample (a LWRHU mount) was placed in the center of a 1.83-m shock tube, and identical charges of CF-8 explosive were placed in each end of the tube. The opposing shock waves were expected to meet at the sample and provide the desired longer impulse. However, the test did not meet its objective and this approach does not seem promising. The safety tests will be performed with the 18.6-kPa·s (2.7-psi·s) impulse.

III. SAFETY TECHNOLOGY

A. Helium Release (D. Peterson, J. Starzynski, and J. Early)

Nine additional samples of $^{238}\text{PuO}_2$ were subjected to heating ramps, and the helium released from them was measured throughout the ramp. The data are being analyzed, and selected samples were submitted for ceramographic analysis.

B. Substoichiometric Fuel (D. Peterson and J. Starzynski)

Attempts to make integral substoichiometric PuO_{2-x} compression samples by sintering cold-pressed bodies at 1900°C were unsuccessful. The samples became substoichiometric, but they did not remain integral. We will attempt to make substoichiometric pellets by cold pressing and sintering mixtures of PuO_2 and carbon. If this method is successful, the properties of the cold-pressed and sintered pellets will be compared to those of the hot-pressed pellets that were tested previously.

TABLE I. Grain Size of the Six Iridium Alloy Capsules Used in the DIT-1 Impact Test.

Capsule ^a	Grain Size (No. of grains across the 0.64-mm wall)	
	Vent Cup	Shield Cup
IRG-101	18-20	20-21
IRG-102	16-18	17-18
IRG-103	17-19	18-19
IRG-104	17-18	19-20
IRG-105	16-17	19-20
IRG-106	19-20	19-21

^aAssemblies IRG-101 through IRG-104 were impacted.
Assemblies IRG-105 and IRG-106 were used for example welds.

TABLE II. Hardness of the Iridium-Alloy Capsules Used in the DIT-1 Impact Test.

Capsule	HARDNESS, DPHN (200 g) (average range)						
	Vent Cup		Shield Cup		Vent	Weld Center	Shield
	Undeformed	Deformed	Undeformed	Deformed			
IRG-101	289	315	315	393	293	308	304
	263-305	283-374	308-325	355-435	292-299	300-314	303-305
IRG-102	319	353	314	393	313	335	316
	309-339	322-391	278-334	351-408	288-332	397-374	301-334
IRG-103	310	379	318	---	315	313	300
	294-333	355-412	299-339	---	290-349	249-349	250-317
IRG-104	335	397	320	390	337	336	323
	313-383	362-441	265-339	364-423	312-407	303-438	301-351
IRG-105	282	---	279	---	278	291	273
	279-297	---	272-285	---	272-283	281-301	267-281
IRG-106	270	---	276	---	266	272	269
	260-284	---	262-290	---	246-282	255-311	254-325

TABLE III. Spectrographic Analysis of GPHS Clads Used in DIT-1.

	IRG-101		IRG-102		IRG-103		IRG-104		IRG-105		IRG-106	
	Vent Cup	Blind Cup	Vent Cup	Blind Cup	Vent Cup	Blind Cup	Vent Cup	Blind Cup	Vent End	Blind End	Vent End	Blind End
Li	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
Be	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
B	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Na	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
Mg	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
Al	50	50	50	50	40	40	30	50	40	80	60	60
Si	10	10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
P	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
K	<3000	<3000	<3000	<3000	<3000	<3000	<3000	<3000	<3000	<3000	<3000	<3000
Ca	3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
Ti	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
V	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Cr	10	<10	<10	<10	<10	20	<10	10	20	<10	<10	<10
Mn	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Fe	100	20	20	20	20	30	<10	10	10	<10	<10	40
Co	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Ni	50	20	40	30	<10	<10	<20	<20	<20	<20	<20	<20
Cu	20	15	15	15	20	30	5	30	10	10	7	15
Zn	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Ga	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Ge	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Sr	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
Zr	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
Nb	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
Mo	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
Ru	<30	<30	<30	<30	—	—	<30	<30	<30	<30	<30	<30
Rh	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Pd	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Ag	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Cd	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
In	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Sm	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Sb	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300	<300
Ba	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3	<3
Hf	<500	<500	<500	<500	<500	<500	<500	<500	<500	<500	<500	<500
Ta	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000	<1000
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%	0.1-1%	0.1-1%	0.1-1%	0.1-1%
Pt	<30	<30	<30	<10	<10	<10	<30	<30	<30	<30	<30	<30
Au	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Tl	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Pb	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
Bi	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30

TABLE IV. Auger Analysis of the Four Iridium Alloy Capsules Impacted in DIT-I.

AUGER RATIOS															
Capsule	Th ₄₉ /Ir ₅₄			Th ₄₉ /Ir ₂₂₉			P ₁₂₀ /Ir ₂₂₉			S ₁₅₀ /Ir ₂₂₉			B ₁₈₀ /Ir ₂₂₉		
	inside	ctr.	outside	inside	ctr.	outside	inside	ctr.	outside	inside	ctr.	outside	inside	ctr.	outside
IRG-101	A1 ^a	0.043	0.062	0.051	0.275	0.443	0.324					0.043			
	A2 ^b	Tr	0.036	0.006	Tr	0.231	0.041								
	A3 ^c	0.050	0.106	0.050	0.355	0.907	0.370							0.150	
Sputtered	A3	0.034			0.268					0.206					
	A4 ^d	0.070	0.083	0.044	0.511	0.784	0.330		0.070?						
IRG-102	A1	0.040	0.104	0.045	0.279	0.800	0.307								
	A2	(Specimen too short)													
	A3	0.056	0.040	0.029	0.333	0.294	0.195								
Sputtered	A3			Tr			Tr			0.085					
	A4	0.038	0.026	0.007	0.300	0.200	0.055		0.073						
Sputtered	A4		Tr			Tr									
IRG-103	A1	0.073	0.069		0.559	0.513									
	A2	0.018	0.081	0.029	0.172	0.583	0.0236								
	A3	0.074	0.076	0.014	0.555	0.638	0.103								
	A4	0.031	Tr	0.044	0.273	Tr	0.362								
IRG-104	A1	0.005	0.071	0.020	0.038	0.528	0.137								
	A2	0.057	0.106	0.042	0.460	0.860	0.321								
	A3	0.056	0.043	0.057	0.415	0.327	0.460								
	A4	0.063	0.074	0.057	0.521	0.640	0.431								

^aA1 = 1 weld.^bA2 = 11 weld.^cA3 = wall, vent end.^dA4 = wall, closed end.

TABLE V. Spectrographic Analysis and Panalysis of DIT-1 Fuel Pellets.

Element	SRP-27 (IRG-101)	SRP-48 (IRG-102)	SRP-6 (IRG-103)	SRP-7 (IRG-104)
Li	<1	<1	<1	<1
Be	<1	<1	<1	<1
B	<1	5 ^a	5 ^a	5 ^a
Na	<2	<2	<2	2
Mg	20	300 ^a	250 ^a	300 ^a
Al	15	60	25	30
Si	85	370 ^a	150	180
K	<5	<5	<5	<5
Ca	50	2000 ^a	3000 ^a	2000 ^a
Ti	15	140	400	160
V	<3	<3	<3	<3
Cr	55	60	65	60
Mn	4	5	5	5
Fe	260	260	340	280
Co	<3	<3	<3	<3
Ni	45	45	50	35
Cu	<1	<1	<1	<1
Zn	5	25	20	25
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	4	30	3	10
Ag	<1	8	5	10
Cd	<10	<10	<10	<10
Sn	<5	<5	<5	<5
Ba	<2	<3	<2	<3
La	<25	<25	<25	<25
Hf	<25	<25	<25	<25
Ta	100	500	100	500
W	<10	<10	<10	<10
Re	<25	<25	<25	<25
Pb	<5	<5	<5	<5
Bi	<1	<1	<1	<1
P(wet)	20	20	20	15
P(radioanalytic)	<20	<20	<20	<20

^aValues exceeding the impurity limits for sintered shards (DPSP-79-1076).

TABLE VI. Sieve Analysis of the Fuel from Capsule IRG-102 after Impact at 58 mps and 930°C.

Particle Size (Micron)	Weight Fraction	Accumulated Weight Fraction
+6000	0.3702	0.3702
+2000	0.3713	0.7415
+841	0.1596	0.9011
+420	0.0426	0.9437
+177	0.0173	0.9610
+125	0.0036	0.9646
+74	0.0062	0.9708
+44	0.0073	0.9781
+30	0.0095	0.9876
+20	0.0033	0.9909
+10	0.0069	0.9978
-10	0.0022	1.0000
≤4	0.0002	

PHASE/TASK	ELAPSED TIME, WEEKS																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
FABRICATE IMPACT MODULE	■	■	■																		
SIMULATED A/S ABLATION MACHINING			■	■																	
OUT-GAS A/S & CBCF				■	■																
MACHINE PG DEPOSIT SURFACES ON GIS	■	■	■	■																	
GIS PG DEPOSITION & MACHINING (PEIZER)	■	■	■	■	■																
OUT-GAS GIS AFTER PG PROCESSING						■	■														
HT CLADS AT MF FOR PuO ₂ ENCAPSULATION	■	■																			
ENCAPSULATE PuO ₂ AT SRP					■	■															
PKG. & SHIP PuO ₂ FC's FROM SRP (2 EACH)						■	■	■													
VENT & ANNEAL PuO ₂ FC HELDS @ LOS ALAMOS									■	■											
ASSEMBLE DIT-1.5 IMPACT HARDWARE										■	■										
IMPACT DIT-1.5 ASSEMBLY											■	■									
POST-IMPACT EXAMINATION												■	■	■							

Fig. 1. Schedule for the DIT-1.5 half-module impact test.

Fig. 2. The partial aeroshell for the DIT-1.5 impact test.

(a)

(b)

Fig. 3. The CBCF sleeve 7787-28-5839 that cracked during outgassing. (a) 1.5x; (b) 10x.

(a)

(b)

Fig. 4. The vent structure of IRG-102 was free of deposits after the 100-h, 1300°C heat treatment. (a) Vent entrance; (b) vent exit, 40x.

(a)

(b)

Fig. 5. Typical microstructures of the walls of capsule IRG-101. (a) Vent cup, 95x; (b) shield cup, 100x.

(a)

(b)

Fig. 6. Typical microstructures of the walls of capsule IRG-102. (a) Vent cup; (b) shield cup, 100x.

(a)

(b)

Fig. 7. Typical microstructures of the walls of capsule IRG-103. (a) Vent cup; (b) shield cup, 100x.

(a)

(b)

Fig. 8. Typical microstructures of the walls of capsule IRG-104. (a) Vent cup, 85x; (b) shield cup, 100x.

(a)

(b)

Fig. 9. Typical microstructures of the walls of capsule IRG-105. (a) Vent cup, 110x; (b) shield cup, 100x.

(a)

(b)

Fig. 10. Typical microstructures of the walls of capsule IRG-106. (a) Vent cup; (b) shield cup, 100x.

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