LA-11274-PR
Progress Report

Space Nuclear Safety Program
January-March 1987

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Space Nuclear Safety Program

January - March 1987

Compiled by T. G. George

SPACE NUCLEAR SAFETY PROGRAM

JANUARY—MARCH 1987

Compiled by
T. G. George

ABSTRACT

This quarterly report describes studies related to the use of $^{238}\text{PuO}_2$ in radioisotope power systems, which were 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 are ongoing; the results and conclusions described may change as the work progresses.

I. GENERAL-PURPOSE HEAT SOURCE (GPHS)/SAFETY VERIFICATION PROGRAM

In November 1985, we completed all safety tests to complement the original NASA Galileo and ESA Ulysses prelaunch safety analyses. The Space Transportation System "Challenger" accident caused significant changes in the Galileo and Ulysses missions. Additional safety tests were needed to account for the new launch configurations, new accident probabilities, and possible changes in the fueled clad characteristics caused by accumulated age before launch.

A. Test Planning/Preparation (R. Zocher, T. Cull)

In this quarter, we continued working on tests that will provide information on the effects of the revised launch configurations and resultant accident scenarios.

We are currently designing three module impact tests and one RTG flyer plate test. We also continued to evaluate the updated accident scenarios to determine if other tests will be required.

B. Bare Clad Impact Test 17 (D. Pavone)

In Bare Clad Impact Test 17 (BCI-17), a simulant-fueled ($^{238}\text{UO}_2$ -depleted) capsule, M-25, was impacted against a concrete target at 75.1 m/s. The target was faced with a 2-cm-thick core of KSC concrete and was reinforced with concrete fabricated from local aggregates. The iridium capsule was fabricated from two unvented iridium cups (0556 and 0073). Before being tested, the capsule was HIPed (hot isostatically pressed) first at 3500 psi and 1000°C for 2 hours and then at 5000 psi and 1000°C for 30 minutes. After being HIPed, the capsule displayed a slight depression on one side (Fig. 1); subsequent radiographic examination (Fig. 2) revealed that this depression traced the contour of a diametric fracture in the uranium pellet.

Postimpact examination of the target revealed that the clad had penetrated approximately 2.0 mm of the KSC concrete (Fig. 3). Although no fractures in the concrete target were apparent, the relatively deep penetration suggests that extensive microcracking did occur.

The iridium capsule was breached by two major and three incipient cracks (Fig. 4). A fuel-fragment push-through occurred 90° to the impact face in the iridium cup that covered the diametric pellet fracture observed

(a)

(b)

Fig. 1. After HIPing, a slight depression was observed on one side of capsule M-25 (a) and (b); 1.5X.

Fig. 2. Radiographic examination of capsule M-25 revealed that a depression in the iridium clad traced the contour of a diametric fracture in the urania pellet.

during radiographic examination (Fig. 4c). A weld centerline crack (Fig. 4d), approximately 16.0 mm in length, also occurred 180° from the major breach. One end of the weld crack was located approximately 4 mm from the "spear point" of the weld overlap. In addition, three small fractures occurred on the radius of the vented cup (Fig. 5). These fractures were apparently related to the differential displacement of the fuel fragment that caused the push-through failure.

The capsule strains, based on pre- and postimpact measurements, were as follows:

Major Diametral = + 7.1%
 Minor Diametral = - 4.5%
 Axial = +11.0%

C. Analyses of Particle Size Data (D. Pavone, R. Zocher)

We reviewed the procedures used to determine particle size distribution in the <10- μ m particle size range in order to identify those factors that contribute to uncertainties in the results.

In the current measurement technique, we record the maximum dimension of the projected image of a particle as it appears in an electron micrograph. In a typical particle size determination, approximately 2000 particles were measured and the number of particles within specific size ranges (such as 0 to 1.0 μ m, >1.0 μ m to 2.0 μ m, >2.0 μ m to 3.0 μ m, etc.) is tabulated. This number is then used to estimate the

Fig. 3. Capsule M-25 penetrated approximately 2.0 mm into the KSC concrete target.

volume fraction of each size range in the total sample; the estimation technique considers all of the particles as spheres with a diameter equal to the size of the largest dimension.

Because the current size-apportioning technique treats all particles as spheres, it conservatively overestimates the actual size of irregular particles. In addition, a size distribution generated by the current method probably underestimates the actual fraction of submicron particles because it is difficult to resolve individual submicron particles that occur in large clusters or agglomerates. Finally, because the sizing technique examines a relatively small number of particles, it is possible that the sample used to generate the distribution is not totally representative.

To determine the amount of error associated with the present sizing technique, we examined electron micrographs of particles obtained from three bare clad impacts. The average particle aspect ratios (major dimension/minor dimension) are given below:

Capsule	Fuel	Mean Aspect Ratio	Sigma
BCI-13	UO ₂	1.62	0.20
BCI-16	UO ₂	1.50	0.22
BCI-15	PuO ₂	1.63	0.17

(a)

(b)

(c)

(d)

Fig. 4.

(e)

Fig. 4. Capsule M-25 was breached by numerous cracks. (a) Impact face, (b) profile, (c) trailing face, (d) opposite profile, and (e) vent end; all at 1.5X.

These results indicate that the UO_2 and PuO_2 particle shapes are roughly equivalent.

To determine the effect of using the maximum particle dimension in the current sizing technique, size distributions for capsule FC-232 (SVT-1) were recalculated using mean particle sizes based on average diameter, average area, and average volume in place of a particle size based on the maximum dimension. The results indicate that the weight fraction of the 0.1- to 1.0- μm particles would have been reduced by 32% if the volumetric mean had been used, by 74% if the diametral mean had been used, and by 95% if the area mean had been employed. At the same time, the 9- to 10- μm particle fraction would have been increased by 18, 32, and 41%, respectively.

D. Investigation of Variables Affecting Fines Generation (D. Pavone, T. Cull)

To increase our understanding of variables that may affect the fraction and distribution of fines in a GPHS capsule, we initiated an experiment to determine the effect of vibration testing. A flight-quality GPHS module was loaded with four UO_2 -fueled capsules, two of which had been HIPed, and was forwarded to Mound for vibration testing.

The vibration testing at Mound was prematurely terminated when an unusually high vibration reading was recorded. The module was returned to Los Alamos and was disassembled. Examination of the module

Fig. 5. Three small fractures occurred on the vent-end radius of capsule M-25; 3X.

(Fig. 6), the graphite impact shells (GISs) (Figs. 7 and 8), and the floating membranes and clads in each GIS (Figs. 9 and 10) did not reveal any anomalous features or evidence of damage.

Although all of the module components appeared to be undamaged, a new GIS with a smaller internal diameter was fabricated to contain the HIPed capsules. We felt that the reduced size of the HIPed capsules,

Fig. 6. The simulant-fueled module returned from Mound was apparently unaffected by the vibration testing.

Fig. 7. GIS 1067, which contained capsules M-6 and M-13, was undamaged.

as well as their resultant loose fit within the standard GIS, may have been responsible for the high vibration reading recorded at Mound. In addition, the outside diameter of the floating membrane was reduced by 15 mils, the GIS cap thickness was increased by 30 mils, and a new aeroshell cap was fabricated to fit the modified GIS. After the new components were fabricated, the module was reassembled and prepared for shipment back to Mound.

Fig. 8. GIS 1087, which contained capsules M-16 and L-21, was undamaged.

Fig. 9. The contents of GIS 1067 were undamaged.

II. LIGHT-WEIGHT RADIOISOTOPE HEATER UNIT (LWRHU) SAFETY

A. Overview

Although all safety tests to complement the original NASA Galileo safety analyses were completed by November 1985, the Challenger accident prompted a significant reconfiguring of the Galileo mission. New safety tests will be needed to account for the new

Fig. 10. The capsules and floating membrane removed from GIS 1087 were apparently unaffected by the vibration testing.

launch configuration, redefined accident scenarios, and potential changes in fuel and cladding characteristics caused by later launch dates and possible orbital maneuvers such as VEEGA (Venus-Earth-Gravity-Assist).

B. Evaluation of Production Heat Treatment (T. Cull, D. Pavone)

A sample of the two-component cement (UCAR cement grade C-34) used to seal the closure plug in the LWRHU aeroshell was prepared, and a portion of the sample was submitted for chemical analysis. The original portion will be outgassed at 400°C with a small platinum-30% rhodium sample to determine what effect, if any, the heat treatment has on the cement and metal.

III. SAFETY TECHNOLOGY PROGRAM

A. Qualification of New Iridium Production Process (T. George)

1. Phase I Tasks. All biaxial testing of new-process and old-process B-batch discs and of Z-batch discs has been completed. The test results indicate that in all of the conditions evaluated, B-batch material was significantly more ductile than Z-batch material. In addition, new-process B-batch material was marginally more ductile than old-process B-batch material. A summary of the test results is presented in Table I. The dispositions of specimens used in the Z-batch and in the new-process/old-process B-batch test series are listed in Tables I, II and III. The chemical compositions of the test specimens are listed in Table IV.

2. Phase II Tasks. We have begun fabricating uranium (^{238}U -depleted) pellets for use in Phase II impact tests. The uranium pellets will be available for encapsulation before the iridium cups have been fabricated.

3. Phase III Tasks. An iridium disc was marked with a circle-grid pattern (Fig. 11) by means of a photoresist/electrolytic polishing technique. The disc has been forwarded to Mound for fabrication into an iridium cup. If the grid is unaffected by the fabrication process, we will use the same technique to mark the cup blanks that will be used in Phase III impact tests.

B. Evaluation of Particle Sizing Technology and Methods (T. George)

In this quarter, we began a comprehensive investigation of particle sizing technology and methods. We undertook this review to resolve the apparent disparity between current particle size analyses and the results predicted by various mathematical formulae, such as a weibull distribution. Primarily, we wanted to determine if any new sizing techniques/technologies have been developed in recent years that might be used to replace the current sieve-and-examine (by means of a transmission electron microscope) sizing technique. If a promising new sizing method could be identified, we planned to purchase the necessary equipment, use both the new and old sizing techniques for the remaining Galileo safety tests, and then switch to the new sizing technique for all particle size analyses after the Galileo mission.

At the beginning of our investigation, we established the following requirements. Any new particle sizing method must

- offer significant improvements over present techniques, both in accuracy and in the number of particles counted;
- minimize the effects of particle agglomeration and of aspect ratio;
- be free of operator error or bias;
- be adaptable to a glovebox environment; and
- be able to analyze a variety of materials.

A subsequent literature search and consultation with other institutions using particle sizing equipment revealed that only the BRINKMAN particle size analyzer fulfilled all of these requirements.

The BRINKMAN particle size analyzer uses a rotating wedge prism to sweep a focused laser beam across a predefined area (approximately 600 μm in diameter) within a sample chamber. The sample chamber may be a standard spectrometry cell (magnetically stirred) or a segment of a flow cell into which a liquid dispersion of particulate material has been introduced. Alternately, the analyzer can be configured to scan the laser beam across the surface of a glass slide onto which a dispersion film has been fixed. In any sample configuration, the analyzer monitors the transmission/occlusion of the laser beam to determine the size of particles within the predefined area.

Agglomerates and particles with poorly defined edges are not counted by the analyzer; only particles with sharp boundaries and maximum diameters between 0.5 and 150 μm are recognized. The analyzer also rejects all particles that are not entirely within the predefined counting area.

Table I. Estimated Plane Strain Failure Limits^a

Material	Temperature (°C)	Nominal Grains/Thickness	Failure Limit (% Major Strain)
Z-batch	1440	5	14
Z-batch	1440	15	53
Z-batch	1440	25	50 ^b
B-batch (old)	1440	25	66 ^b
B-batch (new)	1440	25	71 ^b
B-batch (old)	1100	15	44
B-batch (new)	1100	15	51
Z-batch	1000	15	33
B-batch (old)	1000	15	37
B-batch (new)	1000	15	41
Z-batch	1000	25	47
B-batch (old)	1000	25	55
B-batch (new)	1000	25	57
B-batch (old)	900	15	24
B-batch (new)	900	15	27
B-batch (old)	800	15	17
B-batch (new)	800	15	15 ^c
Z-batch	800	25	32
Z-batch	600	25	11

^aAll tests were conducted in near-plane strain conditions and at a punch velocity of 45 m/s.

^bThe failure criteria applied to this test condition considered localized necking as equivalent to failure.

^cVery few data points exist for this test condition.

Table II. Disposition of Specimens Used in the Z-Batch Test Series^a

Specimen I.D.	Heat Treatment ^b	Geometry	Strain State	Test Temperature (°C)	Average Grains/Thickness ^c
VR227-6	A	disc	+0	1440	23.5
ZR566-3	A	disc	+0	1440	24.9
ZR588-3	A	strip	+,-	1440	23.2
ZR588-6T	A	strip	+,-	1440	16.4
ZR576-1	B	disc	+0	1440	10.0
ZR576-8d	B	disc	+0	1440	11.3
ZR588-4	B	strip	+,-	1440	18.6
QR828-6	C	disc	+0	1440	4.3
VR235-2	C	disc	+0	1440	5.3
ZR572-1	C	disc	+0	1440	5.8
Z563-5d	A	disc	+0	1000	22.8
ZR576-5	A	disc	+0	1000	25.9
ZR579R-5	A	disc	+0	1000	24.3
RR932-2	B	disc	+0	1000	15.3
RR932-6	B	disc	+0	1000	11.5
Z561-6d	A	disc	+0	800	22.5
ZR567-5	A	disc	+0	800	24.9
Z563-2	A	disc	+0	600	20.5
ZR576-3	A	disc	+0	600	25.8

^aAll test specimens were impacted at 45 m/s.

^bThree heat treatments were used to modify the iridium grain size:

A = annealed for 1 h at 1500°C

B = aged for 18 h at 1500°C.

C = annealed for 1 h at 1500°C, then aged for 1 hour at 1800°C.

^cThickness of 0.65 mm.

^dThe disc was not tested to failure; after the first test, it was machined into a 20-mm-wide tensile specimen, which was tested to failure.

Table III. Disposition of Specimens Used in the New-Process/Old-Process Comparison^a

Specimen I.D.	Material	Heat Treatment ^b	Strain State	Test Temperature (°C)	Average Grains/Thickness ^c
B1-1-5	New Proc.	A	+0	1440	22.7
B1-2-6	New Proc.	A	+0	1440	22.3
B701-1	Old Proc.	A	+0	1440	21.3
B701-2	Old Proc.	A	+0	1440	22.7
B2-2-3	New Proc.	B	+0	1100	12.8
B2-8-5	New Proc.	B	+0	1100	11.4
B701-3	Old Proc.	B	+0	1100	13.2
B727-4	Old Proc.	B	+0	1100	16.2
B1-1-1	New Proc.	A	+0	1000	23.3
B1-2-2	New Proc.	A	+0	1000	18.8
B1-7-3	New Proc.	A	+0	1000	19.5
B703-3	Old Proc.	A	+0	1000	28.2
B704-1	Old Proc.	A	+0	1000	22.7
B1-4-2	New Proc.	B	+0	1000	14.0
B1-7-5	New Proc.	B	+0	1000	11.8
B704-3	Old Proc.	B	+0	1000	12.7
B704-4	Old Proc.	B	+0	1000	12.8
B1-4-1	New Proc.	B	+0	900	13.8
B2-1-2	New Proc.	B	+0	900	11.2
B2-8-3	New Proc.	B	+0	900	12.3
B703-2	Old Proc.	B	+0	900	13.5
B732-5	Old Proc.	B	+0	900	13.2
B2-2-4	New Proc.	B	+0	800	14.4
B2-5-1	New Proc.	B	+0	800	14.0
B729-2	Old Proc.	B	+0	800	9.2
B731-1	Old Proc.	B	+0	800	10.6

^aAll test specimens were in the form of discs and were impacted at 45 m/s.^bTwo heat treatments were used to modify the iridium grain size:

A = annealed for 1 h at 1500°C.

B = aged for 18 h at 1500°C.

^cNominal thickness of 0.65 mm.

Table IV. Spectroscopic Analyses of the Biaxial Test Specimens^a

Specimen I.D.	Selected Elements (ppm)								
	Fe	Al	Ca	Ni	Si	Cr	Mo	Cu	Pt
QR828-6	100	60	ND ^b	ND	15	10	ND	25	30
RR932-2	20	40	ND	20	30	40	1000	10	ND
RR932-6	10	60	ND	ND	20	10	ND	10	ND
VR227-6	40	60	3	ND	20	20	1000	20	ND
VR235-2	40	70	3	30	30	100	300	30	ND
Z561-6	30	70	ND	ND	ND	ND	ND	15	ND
Z563-2	30	50	ND	ND	ND	ND	ND	20	30
Z563-5	1000	80	5	ND	ND	10	ND	50	ND
ZR566-3	50	50	3	ND	ND	ND	ND	30	ND
ZR567-5	30	60	ND	ND	10	ND	ND	20	ND
ZR572-1	100	70	3	ND	50	10	ND	50	ND
ZR576-1	50	50	ND	ND	20	10	ND	50	ND
ZR576-3	70	70	ND	ND	30	10	ND	30	ND
ZR576-5	50	100	5	ND	100	15	ND	40	ND
ZR576-8	150	80	3	ND	60	10	300	30	30
ZR579R-5	30	80	ND	ND	60	ND	ND	20	ND
ZR588-3	100	80	3	ND	15	20	ND	60	ND
ZR588-4	30	50	ND	ND	15	ND	ND	60	ND
ZR588-6T	30	50	ND	ND	15	ND	ND	50	ND
B1-1-1	100	80	ND	20	10	20	300	40	ND
B1-1-5	120	80	3	ND	ND	10	ND	50	ND
B1-2-2	150	80	ND	80	ND	30	ND	50	ND
B1-2-6	120	80	ND	40	ND	20	ND	50	30
B1-4-1	80	80	3	50	10	15	ND	25	30
B1-4-2	100	100	3	50	10	15	ND	30	30
B1-7-3	150	100	3	40	ND	15	ND	15	30
B1-7-5	40	60	ND	15	ND	10	ND	10	ND
B2-1-2	30	80	ND	ND	ND	ND	ND	10	ND
B2-2-3	30	100	ND	ND	ND	ND	ND	20	ND
B2-2-4	40	100	3	ND	ND	ND	ND	20	ND
B2-5-1	50	80	ND	ND	ND	ND	ND	15	30
B2-8-3	50	80	ND	10	ND	ND	ND	15	ND
B2-8-5	30	80	ND	ND	ND	ND	ND	6	ND
B701-1	ND	100	ND	ND	30	ND	ND	10	ND
B701-2	ND	150	ND	ND	30	ND	ND	10	ND
B701-3	ND	150	ND	ND	30	ND	ND	10	ND
B703-2	ND	70	ND	ND	ND	ND	ND	10	ND
B703-3	ND	80	ND	ND	ND	ND	ND	15	ND
B704-1	ND	100	ND	ND	ND	ND	ND	15	ND
B704-3	ND	80	ND	ND	ND	ND	ND	30	ND
B704-4	ND	80	ND	ND	ND	ND	ND	30	ND
B727-4	40	100	ND	ND	ND	ND	ND	20	ND
B729-2	50	80	ND	100	ND	10	ND	15	ND
B731-1	50	60	ND	40	10	ND	ND	6	ND
B732-5	50	60	ND	20	ND	ND	300	6	ND

^aElements are listed only if they exceed the detectability limit in at least one specimen.^bNone detected.

Fig. 11. A circle grid pattern was electropolished onto the surface of a DCP-26 iridium disc; 50X.

The BRINKMAN analyzer can be set to count from 1,000 to 30,000 particles. The unit reports all results in terms of number and volume fraction (equivalent to weight fraction) of particles within specific size ranges. Although the unit only considers the maximum diameter of each particle when compiling the results, the sheer number of particles measured and the discrimination built into the measurement system tend to minimize the effects of particle aspect ratio and agglomeration. Since the measurement process itself is not dependent on the use of a particular suspension medium (the only requirement is that it be transparent to the laser beam), the suspension medium can be matched to the physical properties of the particulates to be measured.

In addition to handling large numbers of data points, the BRINKMAN analyzer can also perform in-depth image analysis on individual particles. In this mode, a video camera attachment allows the operator to view particles within the cuvette in real time. When the operator observes either an interesting particle or group of particles, the image can be frozen and stored in memory. After the memory has been stored, it can be enhanced to improve resolution and sharpness. When the image has been sufficiently improved, the analyzer can be used to determine the dimensional characteristics (length, diameter, ferets diameter, center of gravity, etc.) of any single particle or of all the particles in the image.

C. Phase Studies (K. Axler)

In this quarter we continued investigating binary and ternary compounds formed from various combinations of aluminum, iridium, and plutonia. Because the results of previous safety verification tests have indicated that molten aluminum reacts with iridium to form a number of intermetallic compounds, it is important that we understand the chemical and mechanical properties of these compounds.

1. Characterization of the Al/Ir System. A sample of Ir-65 at.% Al was examined by electron microprobe. The specimen had been produced by arc melting the two metals together, grinding the resulting button into powder, pressing the metal powder at 16,000 psi, and then heating the pressed cylinder from 700°C to 1455°C. The microprobe results indicated the presence of the AlIr compound and a second phase of Ir-14.5 at.% Al. These results substantiate that there are no compounds in the Al/Ir system more iridium-rich than the IrAl compound. Although a solid solubility of 14.5% aluminum is slightly higher than we have observed in previous samples, the sample may have been supersaturated with aluminum.

2. Pu/Al/Ir Compounds. A 1:1:1 molar ratio Pu:Al:Ir sample was produced by arc melting all three components into a solid mass. After melting,

the sample was annealed at 1400°C in an argon atmosphere. Subsequent microprobe examination revealed that the sample consisted of two distinct phases. The major phase had an approximate stoichiometry of 1:1:1 Pu:Al:Ir and an orthorhombic crystal structure. The minor phase appeared similar, but with a slightly higher aluminum content. There are no analogies to either phase in the 39 known actinide/group III/transition compounds.

D. Iridium Recovery (P. Kleinschmidt)

We used thermochemical modeling to test if gaseous iridium oxides could be used to decontaminate iridium scrap. We found that reasonable material transfer rates appear to be possible at 1 atm oxygen pressure and 1200°C. Unfortunately, our preliminary studies have indicated that kinetic effects play a large part in the transfer process, and consequently render it too slow to be of practical use. The preferential formation of solid iridium oxide on the metal surface and

the low condensation coefficient of the IrO_3 gaseous species seem to significantly affect the transport process.

We also investigated an alternative transport process that uses iridium halides in place of the oxide species. In theory, iridium halides could be formed at relatively low temperatures and then be disassociated at a hotter surface. The reaction would proceed as follows:

Preliminary experiments with iridium iodides have indicated that the process does work. However, iodine is not a viable transport compound because a significant amount of plutonium is transported along with the iridium. We plan to investigate other halogens to determine if they are as susceptible to the problems as iodine.

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