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Space Nuclear Safety Program

May 1984

Los Alamos Los Alamos National Laboratory
Los Alamos, New Mexico 87545

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Compiled by
T. G. George

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SPACE NUCLEAR SAFETY PROGRAM
May 1984

Compiled by
T. G. George

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 are ongoing; results and conclusions described may change as the work continues.

I. GENERAL-PURPOSE HEAT SOURCE (GPHS)

A. Marine Environment Test (D. Pavone)

1. Test Procedure. A vented, iridium-alloy-clad $^{238}\text{PuO}_2$ pellet was examined after being exposed to a marine environment at the Naval Ocean Systems Center (NOSC); the experiment was under the direction of Dr. H. V. Weiss. The test object, an individual GPHS heat source, is a right circular cylinder plutonia pellet, 62.5 W(t), encapsulated in a 0.64-mm-thick DOP-26 iridium-alloy clad with a sintered, iridium powder vent. During the test exposure, an agglomerate of ocean sediment accumulated around the heat source. When it was shipped to Los Alamos, the clad was evenly coated and was approximately 2.5 in. in diameter.

In order to maintain the mineralogical character of the agglomerate coating, all operations after removal from the test site were carried out at low temperature. The test object was stored on a bed of ocean sediment in a 50-gallon drum filled with seawater, pending shipment to Los Alamos. For shipment, the fueled clad was placed in the primary canister of a DOT-6286 shipping container filled with seawater. A test conducted at Los Alamos had shown that at ambient conditions the water in the primary container would attain a temperature of 52°C in the presence of a 62.5-W(t) heat source. The heat source was stored in the shipping container for 4 months before the postmortem examination began.

2. Postmortem Examination. For the initial operation, we used a stainless steel pan containing enough distilled water to cover the encrusted heat source. When loaded into the shipping container, the agglomerate

coating was free of cracks; however, when the clad was removed from the shipping canister and transferred into a pan of distilled water, several cracks up to about 2.5 mm wide were present. Figure 1 shows four views of the encrusted heat source. A section of the crust about 6 mm thick separated during the handling process, indicating that the crust consisted of at least two layers. The remainder of the outer layer was removed by gently prying with a screwdriver.

The exterior surface of the inner crust resembled the interior surface of the outer crust, although a few narrow cracks were visible (see Fig. 2). Because we did not know whether the crust was tightly bonded to the iridium, we at first separated only a few small samples, approximately 6 × 18 mm, that contained intact cross sections. After a few such sections were carefully sawed and pried loose, we saw that the crust was not bonded to the iridium, so a circumferential cut was made to obtain the remainder of the crust in two large pieces. The crust consisted of a continuous layer of a white material about 3 mm thick; the remaining 7-8 mm consisted of sedimentary particles cemented together with the white substance. When initially exposed, the iridium exhibited a metallic luster; however, the surface tarnished shortly after exposure. Aluminum alloy and brass fixtures immersed in the water at the time of exposure could have caused the surface film through electrolytic action. Figure 3a shows the capsule resting in one of the large pieces of the crust, and Fig. 3b shows the interior of the crust that covered the vent end of the capsule. Plutonium contamination of the crust fragment was quite low, 100-200 counts/min by direct count.

Figure 4 shows the fueled capsule and its bulged ends. The average dimensions of the clad were as follows:

Vent-cup diameter = 30.00 mm (1.181 in.),
Weld diameter = 30.12 mm (1.186 in.),
Shield-cup diameter = 29.95 mm (1.179 in.),
Length (including decontamination cover) = 35.00 mm (1.378 in.).

The diametral dimensions were within the size range of the current GPHS SVT series assemblies. "As fabricated" length dimensions that include the decontamination cover are not routinely obtained, but can be estimated from the design dimensions of the components. The nominal dimensions for the heat source give an original length of 31.43 mm. The change in length is thus estimated to be 3.57 mm or +11.4%.

Only small quantities of ocean salt deposits were observed on the surface of the plutonia fuel pellet. No corrosion of the iridium capsule interior was observed.

A metallographic cross section through the vent assembly (as ground) showed that the space between the decontamination cover and the exterior of the capsule was filled with deposited material. However, the deposit did not survive the polishing operation, only a small portion of it remained on the surface, as shown in Fig. 5a. Figure 5b, an enlarged view of the vent hole, shows a few relatively large, low-porosity particles embedded in a matrix that appears to be imperfectly polished. Electron microprobe analysis indicated that two of the particles contained principally aluminum, silicon, and oxygen, with low concentrations of sodium and calcium. A third particle contained iron, silicon, and oxygen, with minor amounts of magnesium and calcium. The matrix material consisted of calcium, sulfur, and oxygen, with small amounts of sodium, magnesium, aluminum, and silicon. The composition of the matrix material is consistent with the compound calcium sulfate (CaSO_4). In addition to the anhydrous compound, two hydrated compounds are known in the $\text{CaSO}_4/\text{H}_2\text{O}$ system, $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The destruction of the deposit under the decontamination cover was probably due to the volumetric change accompanying hydration of the calcium sulfate.

The iridium microstructure in the capsule walls and vent assembly was apparently unaffected by exposure to a marine environment. No intergranular or bulk corrosion was observed in any of the metallographic specimens.

A ceramographic cross section of the inner crust is shown in Fig. 6. As with the metallographic cross section of the iridium capsule, the inner layer of the crust (CaSO_4) eroded during the polishing operation. Estimated thicknesses are 3 mm for the CaSO_4 and 7-8 mm for the remainder of the crust.

Significant observations of the postmortem examination can be summarized as follows:

(1) During seawater exposure, the capsule was first enveloped by a continuous coating of CaSO_4 about 3 mm thick, followed by accumulation of sedimentary particles cemented by CaSO_4 .

(2) The vent hole of the capsule was plugged with calcium sulfate, causing the ends of the capsule to bulge under pressure exerted by accumulation of helium.

(3) No corrosion of the iridium capsule was observed.

B. Safety Verification Test 6 (T. George)

Details of the test parameters and module disassembly were given in previous monthly reports. In this month we continued with the postmortem examination; we opened and defueled the iridium clads and began metallographic examination of the test components. The fuel in capsule HF-361 was submitted for a particle-size analysis, and samples of the iridium clads and plutonia fuel pellets were selected for chemical analyses.

Capsule HF-361 was transferred to the glove box train used for fines analysis and was opened underwater (to prevent the escape of small fuel fragments). The fuel in this capsule was broken into several large fragments (Fig. 7) that were held tightly by the deformed clad. Following macroscopic examination and photography, the fuel was removed from the capsule halves and prepared for particle-size analysis.

The trailing capsules (HF-189 and HF-225) were photographed, defueled, and packaged for recovery. After defueling, the primary capsules (HF-361 and HF-373) were sectioned to provide specimens for metallographic and chemical analyses.

Preliminary results of the metallographic examinations are summarized below.

1. HF-373. Capsule HF-373 was the only primary clad that breached. The impact face contained a large axial crack (Fig. 8) and a smaller parallel crack on the vent-cup radius. Both cracks apparently resulted from the differential displacement of a large fuel fragment. Cross sections of the main fracture (obtained from the vent and weld-shield cups) show that the fracture was intergranular and that it occurred with a minimum of plastic deformation (Figs. 9 and 10). However, a few elongated grains were observed on the fracture cross section removed from the weld-shield cup (Fig. 9b). The narrow crack on the vent-cup radius had the same intergranular morphology observed in the main fracture (Figs. 11 and 12). Several shallow cracks were present in a reverse bend on the impact face of the weld-shield cup (Fig. 13). These cracks were also intergranular, and they penetrated more than 25% of the wall thickness.

The average grain sizes of the HF-373 and HF-361 iridium-alloy cups are listed in Table I. The HF-373 vent cup contained several areas with enlarged grains (Figs. 14a and 14b). Both the primary fracture (Fig. 15) and the small crack on the vent-cup radius (Fig. 16) occurred in coarse-grained sections. The shield cup,

TABLE I. Grain Sizes of the SVT-6 Primary Clads

	<u>Axial</u>	<u>Transverse</u>
HF-361		
Vent Cup	13.4	18.4
Weld-shield Cup	15.0	12.7
HF-373		
Vent Cup	15.6	13.2
Weld-shield Cup	16.5	16.0

however, was relatively fine grained; sections adjacent to the primary fracture experienced only moderate grain growth (Figs. 17a and 17b).

Apart from the grain growth in the vent cup, the iridium alloy contained only minor defects. A small section of porosity (Fig. 18) and a narrow region of surface delamination (Fig. 19) were observed in sections removed from the weld-shield cup. No internal defects were observed in the vent-cup sections.

The weld in capsule HF-373 was of good quality and contained no defects.

2. HF-361. Although capsule HF-361 did not breach, it was severely deformed by the impact. The impact face contained several sharp bends in the vent and weld-shield cups (Figs. 20 and 21). Numerous small cracks were observed on the exterior of a section removed from the trailing face of the weld-shield cup (Fig. 22). These cracks were very shallow and penetrated less than 10% of the wall thickness.

The vent and weld-shield cups both contained sections of moderate porosity (Figs. 23 and 24). The porosity did not appear to promote cracking or delamination in either cup.

The grain sizes of the HF-361 iridium cups are listed in Table I. Although a minor amount of grain growth occurred in some sections of the vent and weld-shield cups (Figs. 25 and 26), the grain coarsening apparently had little effect on capsule performance.

The weld in capsule HF-361 was of acceptable quality and contained no defects (Fig. 27).

C. Fragment Test (R. Tate)

In this month we completed a second bullet/fragment test on a GPHS half-module fueled with UO_2 simulant pellets. The desired test conditions were a module temperature of 1091°C and a bullet velocity of 457 m/s (1500 ft/s). As before, a .50-caliber, 18-g, 2219-T87 aluminum-alloy bullet was used for the test. To correct the problem of inaccurate impacts, a number of firings

were made before the test, and this problem was thought to be resolved. However, the first bullet fired at the hot module struck the side of the furnace and was deflected into the module at a low velocity. A second bullet was quickly fired at the module when the temperature was 1030°C ; it also struck the furnace and hit the module off center. Figure 28, a posttest photo of the half-module, shows an impact mark on the right side caused by the first bullet and shows the second bullet, melted because of the module temperature, imbedded in the left side. The velocity of the second bullet, measured from a distance on the flash x-ray film, was 415 m/s (1361 ft/s). The bullet struck the fuel capsule tangentially. The view of the impacted capsule (IRG-119) in Fig. 29 shows that the bullet opened the weld seam about 300° around the capsule circumference.

After this test, the inaccuracy of the firings was traced to a worn gun barrel. The barrel was replaced and subsequent firings have given accurate, reproducible results. This bullet test will be repeated.

II. LIGHT-WEIGHT RADIOISOTOPE HEATER UNIT (LWRHU) (R. Tate)

After the .50-caliber gun barrel was replaced and tested for accuracy, two LWRHU units were exposed to .50-caliber, 18-g, 2219-T87 fragment impacts—unit 169 at 757 m/s (2483 ft/s) and unit 013 at 908 m/s (2979 ft/s). The units were fueled with depleted UO_2 and tested at ambient temperature.

The unit 169 capsule was significantly deformed but did not breach (Fig. 30). No identifiable pieces of unit 013 were found in the catch box or in the vicinity. However, graphite debris collected from the catch box was analyzed and found to have significant uranium content. This observation suggests that a fragment at 908 m/s breaches the capsule. The first bullet test performed on the LWRHU was at a velocity of 940 m/s with a similar result; that is, no pieces were found but the graphite debris had a significant uranium content. Apparently, the velocity threshold for breaching a LWRHU capsule with an 18-g, 2219-T87 alloy fragment ranges between 750 and 900 m/s.

In a LWRHU Materials Review Board meeting, the data packages from 51 nonconforming fueled LWRHU capsules were examined. With the concurrence of personnel at the Applied Physics Laboratory, who were the reentry analysts for the LWRHU, the weld-bead stand-off height was reduced from a minimum of 0.75 mm to 0.50 mm. As a result of this change in specification, 30 LWRHU units were accepted as flight-quality units.

III. SAFETY TECHNOLOGY—IRIDIUM BIAxIAL TESTING (T. George)

Twelve 2.50 in. $\times$ 0.375 in. tensile specimens cut from sheet ZR588 were annealed 1 h at 1500°C (for straightening) and were sent to be ground to a uniform 0.025-in. thickness. If the tensile specimens are returned soon, we plan to grid and heat-treat in June and test sometime in early July.

Four biaxial test disks were heat-treated and forwarded to the biaxial test facility; the disks should be tested by mid-June (test details are given in Table II).

Scanning electron microscope (SEM) examination of the disks tested at 1440°C, 1000°, 800°, and 600° (average 25 grains/thickness and impacted at 45 m/s) revealed a number of interesting features.

Disk VR227-6, tested at 1440°C, had necked down to a knife edge (Figs. 31 and 32). The fracture surface was a

thin line interrupted by isolated areas in which a large grain or internal delamination intersected the surface.

The fracture surfaces of disk ZR579-5 (impacted at 1000°C), disk ZR567-5 (impacted at 800°C), and disk ZR576-3 (impacted at 600°C) were very similar (Figs. 33-35). The most noticeable difference between fracture surfaces was the amount of reduction before failure. It is interesting that although the ZR576-3 and ZR567-5 failures (Figs. 34 and 35) were exclusively intergranular, isolated areas of intragranular failure were visible on the ZR579R-5 fracture (Fig. 33).

SEM examination of disk ZR576-5 (impacted at 1000°C, average of 25 grains/thickness) revealed numerous small cracks on the disk surface (Fig. 36). All of the cracks had an intergranular appearance (Fig. 37). Although none of the fractures had gone completely through, many of the cracks appeared to be quite deep. The crack orientations suggest that the grinding marks may act as stress concentrators.

TABLE II. Disposition of Biaxial Test Disks

Disk	Grains/Thickness	Test Temperature (°C)	Velocity (m/c)	Strain State
VR235-2	5	1440	45	+0
Z563-5	25	1000	45	+0
Z561-6	25	800	45	+0
Z563-2	25	600	45	+0

Fig. 1a

(b)

(c)

(d)

Fig. 1. The exposed heat source when removed from the shipping container.

Fig. 2. The exterior surface of the inner crust, showing several small cracks.

Fig. 3a

(b)

Fig. 3. The heat source and inner crust. The encrustation was not bonded to the iridium: (a) clad with portion of inner crust still attached, and (b) interior of crust with clad removed.

Fig. 4. The heat source after the crust was removed. The ends of the heat source were bulged, indicating internal pressurization.

(a)

(b)

Fig. 5. Metallurgical cross section through the vent of the iridium capsule; (a) $\sim 7X$, (b) $100X$. The vent was plugged with a deposit of CaSO_4 and sedimentary particles.

Fig. 6. The inner crust consisted of an interior layer of CaSO_4 about 3 mm thick and an additional 7-8 mm layer of ocean sediment particles bonded with CaSO_4 .

Fig. 7. The fuel in capsule HF-361 was broken into large fragments; 1.5X.

Fig. 8. Capsule HF-373 was breached by a large axial crack and by a smaller crack on the vent-cup radius; 2.0X.

Fig. 9a

(b)

Fig. 10. Cross section of the primary fracture removed from the HF-373 vent cup; 50X.

Fig. 11. Small crack on the vent-end radius of capsule HF-373. As polished, 50X.

Fig. 12. The small crack on the vent end of HF-373 exhibited the same intergranular morphology observed in the main fracture. Etched, 50X.

Fig. 13a

(b)

Fig. 13. Shallow intergranular cracks were present in a reverse bend on the impact face of the HF-373 weld-shield cup. (a) As polished and (b) etched; both at 50X.

Fig. 14a

(b)

Fig. 14 Grain coarsening was observed in several sections of the HF-373 vent cup, 100X.

Fig. 15. A vent-cup section adjacent to the main fracture in capsule HF-373 was relatively coarse grained; 100X.

Fig. 16. Grain coarsening was also observed in sections adjacent to the small crack on the vent-end radius; 100X.

Fig. 17a

(b)

Fig. 17. Sections adjacent to the primary fracture, removed from the HF-373 weld-shield cup; 100X.

Fig. 18. A small area of subsurface porosity was visible on an axial cross section of the HF-373 weld-shield cup. As polished, 100X.

Fig. 19. A section removed from the HF-373 shield-cup radius contained a small delamination. As polished, 50X.

Fig. 20. Sharp bend on the impact face of the HF-361 weld-shield cup; 50X.

Fig. 21. Reverse bend on the impact face of the HF-361 vent cup; 50X.

(a)

(b)

Fig. 22. Cracks in the trailing face of the HF-361 shield cup penetrated less than 10% of the wall thickness. (a) As polished and (b) etched; both at 50X.

Fig. 25. A minor amount of grain growth was observed in the HF-361 weld-shield cup, 50X.

Fig. 26. Grain coarsening occurred in isolated areas on the interior of the HF-361 vent cup, 50X.

Fig. 30. Cross section of the capsule from LWRHU unit 169 after bullet/fragment test at 757 m/s.

Fig. 31. Disk VR227-6 (tested at 1440°C) necked to a knife edge; 100X.

Fig. 32. The fracture surface on VR227-6 consisted of a thin line interrupted by areas in which a large grain or delamination intersected the neck; 500X.

Fig. 33. Isolated areas of intragranular failure were visible on the ZR579R-5 fracture surface (tested at 1000°C); 100X.

Fig. 34. The grains in disk ZR567-5 (impacted at 800°C) were slightly elongated; 100X.

Fig. 35. Disk ZR576-3 (tested at 600°C) failed in a completely brittle manner; 100X.

(a)

(b)

Fig. 36. Numerous cracks were observed on the surface of disk ZR576-5 (tested at 1000°C). Both at 50X,

Fig. 37. All of the cracks on the surface of ZR576-5 had an intergranular appearance; 750X.

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