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by

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PERFORMANCE OF MULTIHUNDRED-WATT FUELED- SPHERE ASSEMBLIES IN THE SAFETY VERIFICATION TEST

by

E. M. Cramer

ABSTRACT

Seven fueled-sphere assemblies equivalent to those proposed for use in multihundred-watt thermoelectric generators were subjected to conditions that simulated the sequential environments produced by an orbital abort and earth impact. The procedures were similar to those in the Safety Sequential Test, and the assemblies differed only in minor dimensional and power level changes. All assemblies met the specifications for flight quality units.

Visual examination indicated that all the iridium shells had lost their containment capability; however, rupturing of two shells was not confirmed. Five were obviously ruptured, and the fuel in three was exposed. All iridium fractures were essentially intergranular. A large grain size may have promoted this type of failure.

Half of the vent assemblies failed to pass helium at ambient temperature after the test. Failure was because of nonmetallic materials in the vent frits.

Release of plutonia per unit area of cracks in a containment shell ruptured by simulated earth impact was determined.

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I. INTRODUCTION

The ability of prototype fueled-sphere assemblies (FSAs) for the modular multihundred-watt (MHW) radioisotope thermoelectric generator (RTG) to contain their plutonia fuel during a simulated orbital abort resulting in atomospheric reentry and earth impact was investigated in a Safety Sequential Test (SST).¹ Upon completion of the SST, emphasis in the Safety Verification Test (SVT) of the heat source hardware shifted from development, during which some specification changes were made, to proofing of the flight-qualified components.

The MHW RTG contains 24 FSAs. Each FSA is a ²³⁸PuO₂ sphere, about 37.21 mm (1.45 - 1.48 in.) in diameter, encapsulated in a vented iridium postimpact containment shell (PICS), 0.57 - 0.71 mm (0.022 - 0.028 in.) thick. This is the basic unit of containment, and it will, after impact, be identified as the postimpact sphere assembly (PISA). Each sphere

assembly is, in turn, enclosed in an 11.81-mm (0.465-in.) -thick graphite impact shell (GIS) of carbon impregnated Thorne[®] yarn. The 24 FSAs are assembled into an aeroshell that protects the heat source during reentry. After reentry, the aeroshell remains and the GIS protect the individual sphere assemblies during impact and leave the noble metal PICS to contain the plutonia until the debris can be recovered.

Mission accident analysis has shown that FSAs will not strike the earth singly; rather, impact will always involve the essentially intact heat source. Practical limits of testing capability preclude using heat sources fully loaded with radioisotopic fuel. However, single FSAs can be tested safely, and, as

¹A product of Union Carbide Corporation, Carbon Products Division, P. O. Box 849, Greenville, SC, 29602.

the mission safety guideline requires demonstration of single FSA performance, the FSA became the test unit although its response would differ from the synergistic response in the heat source.

Iridium for the containment shells was from three sources. Types WC-n and WE iridium were produced from two separate batches of powdered metal by Oak Ridge National Laboratory* and furnished as sheet. The material had been electron-beam melted and contained a nominal 0.3% tungsten. Type III iridium, a product of Englehard Industries Inc.,** was furnished as sheet or as formed hemispherical shells. The material had been arc melted and also contained a nominal 0.3% tungsten. Mound Laboratory formed the sheet material into hemispherical shells (PICS). To increase the strength of the flight unit PICS, wall thickness had been increased about 25% over that used in the SSTs, from 0.508 to 0.56-0.71 mm (0.021 to 0.022-0.028 in.).

The vent design also was changed. In the SST vent, gas flow was through a central port in a shield disk, across both surfaces of an iridium-filter disk sandwiched between the shield disk and the PICS, and, finally, through a 0.127-mm (0.005-in.) port in the PICS to the interior of the heat source. In the SVT vents (see Fig. 31), the iridium frit is sandwiched between two disks. Gas flows through an annular port in the shield disk, through the frit to a central cavity in the base washer, and, finally, through a 0.127-mm-diam port in the PICS to the interior of the heat source. The new design presents a much greater frit area to the outflowing gas than did the SST type which normally became blocked by non-metallic material deposited just inside the shield disk port.

Each FSA's power level was increased from 100 to 102 thermal watts at the beginning of the mission, and the outside of the PICS was grit blasted to enhance emissivity and thus maintain, or lower, the FSA operating temperature. Further, the fuel spheres for the SVT were given an additional oxidation treatment before assembly, because evidence of substoichiometry had been seen in SST fuels.

II. TEST PROGRAM

The test program was intended, like the SST, to simulate the sequence of environments experienced

by a live FSA during an aborted mission. The ultimate purpose was to determine FSA response for safety analyses and to define deviations from the intended performance of the flight-qualified assemblies. The environments included normal handling, a standard vibration test, aging for at least 2.6 Ms (30 days) at operating temperature, both maximum and minimum reentry heat pulses, and worst-case earth impact onto granite. The test plan³ specified the parameters discussed below.

Eight multihundred-watt fueled test (MHFT) FSAs were fabricated to production specification by Mound Laboratory and vibrated to the acceptance level specified by the prime contractor.* The eight units were shipped to Los Alamos Scientific Laboratory (LASL) to be aged for at least 2.6 Ms at a GIS surface temperature of 1210° C, the temperature of a grit-blasted FSA in an orbiting heat source, as compared with 1268° C specified in the SST. This lower temperature was intended to reduce vapor transport of plutonia and tramp impurities in the FSAs.

The reentry heat pulses were to correspond to either profile in Fig. 1, which shows the GIS temperatures in the LASL impact gun which should give a PICS temperature equal to the average profile of all the PICS in a heat source reentering under best (cold) and worst (hot) credible conditions.

Impact temperatures were to be either 1100 or 1440° C, depending on the reentry pulse, and the

*General Electric Company, Isotopes and Systems Application Operation, P. O. Box 8661, Philadelphia, PA, 19101.

Fig. 1.
SVT reentry temperature profiles.

*New Hollifield National Laboratory.

**Englehard Industries, Inc., 700 Elair Road, Carteret, NJ, 07008.

velocity was to be 85 ± 2 m/s. The velocity is about 10% more than the SST velocity, as flight-qualified units were involved. Figure 2 shows the test sequence.

III. PROCEDURES

The verification test essentially paralleled the sequential test in scope and purpose, so the same equipment was used. As the equipment was described in the SST summary report,¹ it is mentioned here only briefly as necessary. Testing included the procedures listed below. All were completed as proposed except the low-temperature impact of one Type WC FSA. This FSA's vents were closed after aging, so it was examined destructively without further treatment, as shown by the closed branch in Fig. 2.

A. Pretest inspection

1. Perform radiography.
2. Measure the external dimensions of the impact shell.
3. Weigh the FSA.

B. Short-term aging

1. Heat treat the FSA in a vacuum at a nominal 1210°C , measured optically on the GIS surface, for at least 2.6 Ms.

Fig. 2.
SVT sequence.

C. Post-aging inspection

1. Perform radiography.
2. Weigh and measure the FSA.
3. Check the vents by helium release during self-heating.

D. Impact test

1. Weld the FSA into an impact can.
2. Subject the FSA to a reentry thermal pulse in the impact gun.
3. Impact on a granite target.

E. Post-impact examination and disassembly

1. Recover the PISA from the impact debris.
2. Collect the GIS debris for total plutonium analysis.
3. Inspect the PISA visually for ruptures and take documentary photographs.
4. Disassemble the PISA.
 - a. Defuel the PICS.
 - b. Check the vents in a bubble test.*
 - c. Examine the PICS metallographically.
 - d. Analyze the plutonia fines
 - e. Examine the plutonia metallographically.
 - f. Take documentary photographs of significant effects.

Because of the similarity of the specimens and test conditions, the effects differed mostly in degree. Consequently, only typical examples are discussed here unless a unique result warrants attention. Each example is identified by MHFT number so that the effect can be related to the specific material and test condition that produced it.

The iridium metallographic specimens were polished mechanically with alpha alumina on nylon cloth in a vibratory polisher and finished (by hand) with 1-mm diamond on a high-napped synthetic cloth. The polished surfaces were etched electrolytically in a solution of 20% hydrochloric acid and 80% water saturated with NaCl at 7.5 V ac. The plutonia metallographic specimens were pressure potted in epoxy resin, ground through 600-grit silicon carbide paper, and polished mechanically with alpha alumina on nylon cloth in a vibratory polisher. The polished surfaces were etched chemically in a solution of 10 ml of hydriodic acid, 10 ml of hydrochloric acid, and about 1 ml each of nitric and hydrofluoric acids.³

*Vents were checked by observing bubbles in Leak-Tek 65-C (a product of American Gas and Chemicals, Inc., New York) applied to the vent shield area while the vent was held manually against a rubber orifice through which helium was passed from a 138-kPa (20-psi) source.

In some instances, a microhardness traverse of a metallographic sample is shown. These values were obtained with a 136° diamond pyramid indenter at 50-g load. Each value is shown in the approximate location of the traverse.

IV. POSTTEST EXAMINATION

MHFT 39 was one of three assemblies aged for 7.2 Ms (80 days) and the first to be impacted after cold reentry (see Fig. 2). Radiography of the assembly upon its receipt at LASL had shown that the plutonia was badly cracked and the sphere was broken after vibration and shipping.

After the aging, a slight white deposit appeared on the GIS surface, but there seemed to be no other heat treatment effects. Further disintegration of the plutonia could not be detected. The PISA was removed from the graphite shell for a vent check* and, in the process, the threads on the graphite cap were damaged. Because a GIS was available from MHFT 41, it was substituted for the damaged one. The vent check showed that at least one vent was open.

The FSA was impacted on granite at 86.56 m/s and 1100° C opposite the GIS cap. An approximately 90° cone of material in the body of the impact shell was crushed, and the cap was delaminated. The PISA deformed fairly regularly, although there was evidence of large fragments of plutonia being present at impact. The PICS (Type WC) had ruptured on all of its surfaces. There were six hoop-type cracks in the periphery, and two tensile tears in the back (Fig. 3) and many more in the impact face (Fig. 4). The PICS also had the most vapor-deposited iridium of any SVT specimen (see Fig. 29).

The plutonia was friable and had fractured mostly into small fragments with many fines. It was small-grained, but the largest grains and the greatest pore volume were in the equatorial zone. Analyses for total plutonium showed that the GIS in which the PISA was aged contained 8 μ g of plutonium (as PuO₂), whereas that in which it was impacted contained 0.72 mg.

MHFT 40 also was aged for more than 7.2 Ms and was the first impacted after hot reentry. Initial radiography had indicated that the plutonia fuel sphere was badly cracked but still intact after vibration and shipping.

After the aging, an unidentified white deposit appeared on the GIS surface, but no other external

Fig. 3.
The PISA from MHFT 39 after impact. Two tensile tears occurred in the back of the assembly.

effect of the heat treatment was visible. Radiography, however, showed that the fuel sphere was no longer intact (see Fig. 19). A check made without disassembling the FSA showed that at least one vent was open.

The assembly was impacted on granite at 68.18 m/s and 1440° C, opposite the impact shell cap. A less than 90° cone of material in the impact shell was crushed, and the body did not split open. The cap, however, delaminated into three pieces.

The PISA deformed fairly regularly (Fig. 5), although there was evidence that three large pieces of plutonia had been present at the time of impact. There seemed to be one small rupture (Fig. 6) in a deep fold in the impact face. There were no fractures in either the back or the periphery of the Type WC PICS (see Fig. 26), nor was there fingerprint cracking in the impact face.

Fig. 4.
Many cracks occurred in the impact face. The longest crack crossed most of the impact face; however, the severest cracking was in the periphery.

*It was agreed that vent checks by helium release in the SST Series would be made with the PISA bare. Subsequent checks in the SVT Series were made with the FSA intact.

Fig. 5.

MHFT 40 after impact. There were no ruptures in the back surface of the PISA.

The plutonia was friable and had fractured mostly into small fragments with a greater than normal quantity of fines. It was small grained. The largest grains were in the equatorial zone, but the greatest pore volume was at a pole where it normally occurs.

MHFT 41 (Fig. 7) was aged for more than 7.2 Ms and was the only FSA destructively examined without reentry or impact (Fig. 21). Its initial radiography had indicated that the plutonia was badly cracked after vibration and shipping but that the fuel sphere was intact.

As in all the SVT assemblies, a slight white deposit formed on the GIS during aging, but no other effects of the heat treatment were noted. The fuel sphere did not seem to have disintegrated further. A helium release check showed that both vents were closed.

Both vents were clean and bright externally. Internally, a heavy plutonia coating covered the PICS (see Fig. 23). The closure weld shield had been dis-

Fig. 6.

There seemed to be a rupture about 1 mm long in the impact face. However, penetration of the iridium shell was not verified in metallography.

Fig. 7.

MHFT 41 after aging. Both vents were closed, and the PISA was destructively examined without impact.

placed about 60° (its microstructure is shown in Fig. 25). There seemed to be no vapor-deposited iridium in its shadow, although a maximum of 10 μ m was later seen in a metallographic section of the wall. Metallography of the vents showed that they were blocked by a combination of plutonia; a complex glassy material containing mostly calcium, silicon, and oxygen with plutonium, iron, and magnesium or aluminum; and sintering, in the filter frits (see Figs. 31 and 32). The Type WC iridium did not appear to have been degraded, and both its hardness and purity were normal.

The plutonia had fractured into mostly large pieces with few fines. Its grains were uniformly small. The lowest pore volume occurred in the equatorial zone of the fuel.

MHFT 45 was the fourth FSA impacted after hot reentry. Its initial radiography had indicated that the plutonia was badly fractured and the fuel sphere was broken after vibration and shipping (see Fig. 18).

As in all SVT assemblies, a slight white deposit formed on the GIS during aging, but no other effects were noted. The fuel sphere did not appear to have disintegrated further. A helium release check after the aging showed at least one vent open.

The FSA was impacted on granite at 85.1 m/s and 1440° C opposite the cap. The impact shell body was crushed and delaminated, as was the cap, and both were jammed tightly in the impact can (Fig. 20). The PISA deformed fairly regularly, although large plutonia fragments obviously had been present at impact. The PICS (Type III) had a small crack about 1 mm long in the back (Fig. 8) at the edge of a fuel fragment and a fingerprint pattern of cracks (Fig. 9) in the impact face (see Fig. 24). All these defects appeared superficial in subsequent

Fig. 8.

MHFT 45 after impact. A small crack occurred in the back of the PISA.

metallography. Typical microstructures in the closure weld are shown in Fig. 28.

The plutonia had fractured mostly into large pieces with some of moderate size and a moderate amount of fines. The material was uniformly fine-grained; however, in this instance, the greatest pore volume was in the equatorial zone. Photomicrographs of the plutonia (Fig. 42) illustrate typically the formation of helium bubbles from spontaneous fission although the effect was not confined to this specimen. An analysis of the GIS for total plutonium showed that it contained 460 μg as the dioxide after impact.

MHFT 47 was the second FSA impacted after hot reentry. Initial radiography had indicated that its fuel was badly cracked and that the plutonia sphere was broken after vibration and shipping.

As in all SVT assemblies, a slight white deposit formed on the GIS during aging, but no other exter-

Fig. 9.

Fingerprint cracking occurred in much of the impact face.

nal effects were noted. The fuel did not seem to have disintegrated further, and a helium release check showed that at least one vent was open.

The FSA was impacted on granite at 85.8 m/s and 1440° C opposite the GIS cap. The graphite body split, and the cap delaminated. An approximately 90° cone of material in the impact face was crushed but remained in place. The whole assembly was loose in the impact can after impact.

The PISA deformed irregularly, and the PICS (Type WE material) impact face ruptured (Fig. 10). The rupture, about 25 mm long and 2 mm wide, occurred at the edge of a large plutonia fragment, exposing the fuel. A small crack in the back (Fig. 11), later shown to be superficial, occurred in a discolored spot where reaction with an unidentified impurity made a shallow penetration in the iridium (see Fig. 38).

The plutonia had fractured into moderate-size fragments with a few large pieces and few fines. The material was uniformly fine-grained; however, pore volume varied from 14% at a pole to 25% in the middle of the sphere. The microstructure, considered typical of the fuels, is shown in Figs. 39-41. The large rupture in the PICS allowed 18.7 mg of plutonia to escape into the impact shell debris.

MHFT 48 was the third FSA impacted after hot reentry. Its initial radiography had indicated that the plutonia was badly fractured and the fuel sphere was broken after vibration and shipping.

As in the other SVT assemblies, a slight white deposit formed on the GIS during aging. The fuel sphere seemed to have disintegrated further in the process. A vent check by helium release was positive.

Fig. 10.

MHFT 47 after impact. A large tear, about 25 mm long and 2 mm wide, exposed the plutonia in the impact face. A metallographic section of the vent assembly is shown in Figs. 33 and 34.

Fig. 11.

A small crack, about 2 mm long, occurred in the back of the PISA. The darkened area is a zone of reaction with an unidentified impurity where shallow grain boundary penetration took place (see Fig. 38).

The FSA was impacted on granite at 87.0 m/s and 1440° C. Although it was meant to strike opposite the GIS cap, the assembly was accidentally rotated about 120° in its container and struck on the threaded joint. The entire graphite body was badly crushed and delaminated, and essentially no free space was left in the impact can.

The PISA deformed both irregularly, because of large fuel fragments, and asymmetrically, the greatest distortion being near the GIS cap. The PICS (WE material) periphery suffered two large hoop type ruptures (Fig. 12) that exposed the plutonia. These ruptures, about 15 and 20 mm long by 2 mm wide, extended from the impact face into the back. They occurred near the point of greatest distortion

Fig. 12.

MHFT 48 after impact. Ruptures originating in the periphery extended into the back of the PISA.

(Fig. 13) and also reflected the presence of large fuel fragments. Some fingerprint cracking occurred in the impact face, but it was superficial. Both vents were closed to helium. Iridium, apparently debris from drilling, partially blocked one vent port, but the cause of failure was an accumulation of non-metallic material, particularly plutonia, in the frit (see Figs. 36 and 37).

The plutonia was friable and had fractured into small fragments with a few moderate-sized pieces and many fines. Its grains were small and uniform, but its porosity, although somewhat obscured by the fuel's having been crushed, was least in the middle of the sphere. The large ruptures in the PICS had allowed 14.7 mg of plutonia to escape into the impact shell debris.

MHFT 49 was the second FSA impacted after cold reentry. Its initial radiography had indicated that the plutonia was badly fractured after vibration and shipping but that the fuel sphere was intact.

As in other SVT assemblies, a slight white deposit formed on the GIS during aging. The fuel sphere was broken, but it may have been broken before aging and not have been detected because of a different orientation in the initial radiography. A vent check by helium release was positive.

The FSA was impacted on granite at 87.3 m/s and 1100° C opposite the GIS cap. Both body and cap of the impact shell were delaminated and, although the assembly was loose in the impact can, the crushed material in the impact face remained in place (see Fig. 21).

The PISA deformed irregularly by stretching over large plutonia fragments (Figs. 14 and 15). The PICS (Type WE material, shown in Fig. 27) was ruptured on all surfaces, mostly at the edges of fuel fragments, but also by fingerprint cracking in the impact face.

Fig. 13.

The large tears were near the point of greatest distortion, at the GIS cap.

Fig. 14

MHFT 49 after impact. The back of the PISA ruptured at the edges of large plutonia fragments

The cracks were up to 12 mm long, but the fuel was not visibly exposed.

The plutonia had fractured into moderate-size pieces with a few small fragments and many fines. Its grain size range was somewhat greater than usual, but pore volume was uniformly distributed among the polar, equatorial and middle regions. The many cracks in the PICS allowed 160 μg of plutonia to escape into the impact shell debris.

MHFT 50, the fifth FSA impacted after hot reentry, was the last of the series. Its initial radiography had indicated that the plutonia was badly fractured and the fuel sphere was broken after vibration and shipping.

Aging was delayed for several months, partly because of an operational failure in the aging fur-

Fig. 15.

The impact face also ruptured in the same manner. A fingerprint crack pattern covers about one third of the face.

nace. This FSA, aged alone using new furnace elements, also had a slight white deposit on its surface. The fuel sphere did not seem to disintegrate further during heat treatment, and a vent check by helium release was positive.

The FSA was impacted on concrete at 85.7 m/s and 1440° C. Impact was meant to be opposite the GIS cap, but the assembly was accidentally rotated in its container so impact was almost side-on (see Fig. 22). Both body and cap of the GIS were crushed and delaminated, but the debris was held tightly in the impact can.

The PISA deformed irregularly (Fig. 16) over approximately a hemisphere of plutonia and asymmetrically, the greatest distortion being near the cap. The type III PICS was ruptured in the back, exposing the fuel. Although the failure was intergranular, the material was ductile (see Fig. 30). The closure weld was cracked in tension in the impact face, and a small area of fingerprint cracks also occurred there (Fig. 17). These cracks were all superficial.

One of the vent assemblies was closed to helium at room temperature. A microsection, Fig. 35, indicated that the vent had not been drilled completely through the wall; however, a dark gray deposit around the port, containing iridium, iron, and tungsten, indicated that it had once been open.

The plutonia was friable. It had broken up into mostly small fragments with many fines. Its grains were uniformly small, and its pore volume was fairly uniform, the greatest density being in the equatorial zone. A total of 1.25 mg of plutonia had escaped the PICS and been deposited in the impact shell debris.

Fig. 16.

MHFT 50 after impact. The PICS was ruptured in the back. The cap axis of the impact shell is at approximately 90° to the crack.

Fig. 17.

Fingerprint cracking and a tensile crack in the closure weld occurred near the top of the impact face.

V. TABLES

Tables I-V are compilations of the information obtained in the Safety Verification Test. The information presented in the previous sections is included, but the Tables are more complete and allow easier comparison of individual samples.

TABLE I

TEST HARDWARE AND PRE-LASL HISTORY

	39	40	41	45	47	48	49	50
FUEL SPHERE ^{a, b}	PPS 42	PPS 43	PPS 44	PPS 132	PPS 120	PPS 13*	PPS 63	PPS 139
Oxide wt (g)	256.0	250.4	250.1	256.4	256.8	256.1	251.5	256.7
Pu wt (g)	217.6	218.6	218.6	218.0	223.5	222.8	217.8	223.3
Pressing date	7/23/73	7/24/73	7/25/73	2/11/74	1/22/74	2/7/74	8/23/74	2/21/74
Density (" theoret.)	81.47	81.96	81.12	84.21	82.72	85.08	86.33	84.03
Outgas date	7/27/73	7/25/73	7/26/73	2/12/74	1/22/74	2/11/74	1/31/74	2/26/74
Diameter (mm) pole	36.86	36.83	36.88	36.80	37.13	36.75	37.01	37.01
equator	37.03	37.03	37.06	37.24	37.34	37.01	37.41	37.13
CONTAINMENT SHELL ^{a, b}	WC-n			Type III	WE		Type III	
Type of iridium	50-1			1198	1-2		1244	
S/N male hemishell ^c	Lot 70			Lot 86	Lot 376		Lot 93	
female hemishell	58-4			1186	2-1		1251	
	Lot 74			Lot 86	Lot 376		Lot 94	
Preassembly HT time (h)	1							
temp. (°C)	1500							
Vent S/N male	---	---	---	P9	97	99	101	93
female	---	---	---	90	98	100	102	94
Washer S/N male	---	---	---	65	87	63	94	91
female	---	---	---	61	92	85	98	68
Decon disk male	---	---	---	395	403	405	407	399
female	---	---	---	396	404	406	408	400
Weld shield	---	---	---	WC-46-10	WC-46-13	WC-46-32	WC-46-17	WC-46-12
Weld date	1/17/74			4/9/74				
Feed width (mm)	---	---	---	2.39/2.44		2.39/241	2.39	2.36/2.44/2.44
Alpha count (dpm)	---	---	---	52/108		134/132	190/120	300/14
before/after leak ck	---	---	---	26/50				
Vibration test limits	Acceptance schedule							
temp. (°C)	1000							
Decon disk opened	---	---	---	4/10/74	4/9/74	4/10/74		
IMPACT SHELL ^{a, b}	836233-1	886224-2	886224-1	885761-1	885759-2	885760-1	885762-1	885761-2
S/N shell	Threaded cap							
Type	2/17/74							
Assembly date				4/10/74	4/9/74	4/10/74		
Alpha count (dpm)	---	---	---	0	14	6	10	2

^a Monthly Newsletter, Nuclear Operations, Mound Laboratory, June 1, 1974.

^b SVT Quality Control Package, E. W. Johnson, MRC, to R. N. R. Mulford, LASL, May 6, 1974.

^c Serial number of the PICS component.

TABLE II

PRETEST INSPECTION

	MHFT 39	40	41	45	47	48	49	50
Received at LASL	3/23/74			5/15/74				
Radiography (FSA)	4/2/74			7/1/74				1/20/75
	PuO ₂ badly fractured							
	Broken		Intact		Broken		Intact	
	No other defects detected							
GIS diameter (mm)	64.87	64.80	64.85	64.87	65.00	64.90	64.90	64.85
	64.85	64.86	64.82	64.81	64.86	64.85	64.85	64.78
Weight (g) (FSA)	449.6	448.3	451.4	451.4	460.4	457.9	458.0	453.9
Appearance (FSA)	Clean and bright							

TABLE III

SHORT-TERM AGING

	MHFT-39	40	41	45	47	48	49	50
Time (h)	2009 ^a	2009	2009	720	720	720	720	731
Temperature (°C)	1207	1207	1207	1214	1214	1214	1214	1205
Appearance (FSA)	Slight white deposit, no other effect noted							
Diameter (mm) pole (FSA)	64.92	64.80	64.92	64.90	64.97	64.85	64.92	64.86
equator (FSA)	64.87	64.87	64.86	64.83	64.85	64.82	64.85	64.90
Weight (g) (FSA)	451.2	448.3	449.8	451.0	460.4	457.9	458.9	453.8
Diameter (mm) pole (PISA)	40.31 ^b	d	40.49 ^c	---	---	---	---	---
weld (PISA)	40.81	d	40.75	---	---	---	---	---
45° (PISA)	40.57	d	40.58	---	---	---	---	---
Weight (g) (PISA)	322.8	d	323.6	---	---	---	---	---
Vent check	At least one open							
Interior of GIS	Clean	d	Clean	---	---	---	---	---
PuO ₂ in GIS (μg)	8	d	e	---	---	---	---	---
Radiography (FSA)	PuO ₂ badly fractured							
Fuel sphere	f	Broken	Intact	Broken				
Cf as rec'd	Worse		Comparable			Worse		Comparable
Clearance PuO ₂ /lr (mm)	Not taken			1.0		1.5	1.0	Not taken

^a Impact conditions for the series had not been determined when the scheduled 30 days of aging was reached. Rather than terminate the treatment and allow helium to build up in the fuel, aging was continued until the information had been received.

^b The GIS was damaged when the PISA was removed for a vent check. It was used for a total plutonium analysis and the test was continued with the GIS from FSA 41.

^c FSA 41 was selected for launch abort simulation because it had closed vents. Its GIS was substituted to FSA 39.

^d Because of possible damage to the GISs as in the case of FSA 39, the remaining FSAs were not opened for vent checking.

^e The GIS was substituted to FSA 39.

^f The FSA position shifted during radiography. Further effects were indeterminate.

TABLE IV

IMPACT TESTING

	MHFT 39	40	45	47	48	49	50
Date	7/15/75	7/10	9/3	8/28	8/30	8/26	1/22/75
Reentry profile	Cold	Hot				Cold	Hot
Velocity (m/s)	86.5	68.2	85.1	85.8	87.0	87.3	85.7
Temperature (°C)	1100	1440				1100	1440
Target	Granite						Concrete
Intended orientation	180° to cap						
Actual orientation	180° to cap				On threads	180° to cap	~80° to cap axis

TABLE V

POSTTEST EXAMINATION

	MIIFT 39	40	41	45	47	48	49	50
IMPACT CAN								
Deformation mode	Symmetrical	---	---	Sym.	Asym.	---	Sym.	Asym.
Failure mode	None. Debris confined	---	---	None. Debris confined	---	---	---	---
IMPACT SHELL (GIS)								
Failure mode								
Body	Not split	---	---	Crushed and delaminated	---	---	---	---
Cap	Delaminated	---	---	Delaminated	---	---	---	---
Relation to can	Assembly loose in can	---	---	Tight in can	---	---	Loose	Tight
PuO ₂ in GIS (μg)	--	15	---	460	18.7 mg	14.7 mg	160	1.25 mg
CONTAINMENT SHELL								
Deformation mode	Fairly regular	---	---	Fairly regular	Irregular over PuO ₂	Fairly regular	Irregular over PuO ₂	---
Diameter max. (mm)	43.61	42.75	---	44.86	44.02	46.43	43.43	46.00
min.	42.42	41.66	---	43.05	42.21	45.67	41.99	44.60
height	31.32	31.78	---	28.65	29.57	28.78	31.37	30.56
Failure mode	Ruptured in sur- faces.	Finger- print ^a	---	Finger- print, Push Through ^a	Ruptured IF and back PuO ₂ ex- posed.	Hoop rupture. Finger- print. PuO ₂ ex- posed.	Ruptured all sur- faces	Ruptured IF and back PuO ₂ exposed.
Tensile cracks - back	2	0	---	1	1	0	6	1
Length/width (mm)	5/0.5	---	---	1/---	2/0.5	---	to 12/0.5	12.5/0.5
Tensile cracks - hoop	6	0	---	0	0	2	4	0
Length/width (mm)	to 12/1	---	---	---	---	to 20/2	10/0.5	---
Tensile cracks - Impact face	Many	1 ^a	---	0	1	0	3	0
Length/width (mm)	to 18/1	1/0.2	---	---	25/2	---	to 10/0.5	---
Fingerprint - IF	No	---	---	Yes	No	Yes	---	---
Cracks - closure weld	No	---	---	No	---	---	1 ^b	1
Type of failure - Tensile Fingerprint	Intergranular	---	---	Intergranular Inter + transgranular	---	Not sectioned	Intergranular	IG; ductile
INTERIOR OF PICS								
CVD iridium thickness (μm)	Present	To 20	To 10	To 10	<10	<10	To 10	<10
CVD plutonia thickness (μm)	Present	To 150	To 80	Indeter- minate	---	---	To 30	Indeter- minate
General strain	Yes	No	---	---	---	Yes	No	---
VENTS								
Bubble test - back ^c	Open	---	Closed	Open	---	Closed	---	---
Open impact face	Open	V sl open	Closed	---	Ruptured ^d	Closed	Open	---
Cause of blockage	PuO ₂	---	---	---	---	---	---	---
	CaSiO w	---	---	---	---	---	---	---
	Fe, Mg, Al	---	---	---	---	---	---	---
Weld shield displacement	~ 80°	90°	~ 60°	~ 90°	---	---	~ 30°	80°
PICS								
Microhardness ^f (VHN)								
Impact face ^c	350-450	320-370	330-370	360-400	310-400	320-430	380-480	330-380
Back	360-490	310-380	320-340	350-380	320-430	320-360	330-420	350-400
Closure weld	320-390	310-400	320-340	330-360	310-350	330-370	360-390	320-350
Weld shield	320-350	Not taken	300-330	Not taken	350-370	Not taken	320-370	310-380
Grain size (μm) PICS	1 to 3 in wall thickness	1 to 2	1 to 2 in wall thickness	2 to 4 wall thickness	1 to 3 in wall thickness	1 to 2 wall thickness	1 to 4 in wall thickness	4 to 5 wall thickness
Weld	200-300 μm	To 300 μm	To 300 μm	To wall thickness	30 to 400 μm	80 to 250 μm	1 to 4 in wall thickness	60 to 100 (μm) wall thickness
Selected impurities ^g								
Al maximum (ppm)	<20	20	<50	<30	ND	---	---	---
Si	100	---	40	<30	20	30	<30	100
Fe	300	---	---	---	500	---	<300	300
Ca	100	50	30	<100	10	20	<100	5

TABLE V continued.

	MHFT 39	40	41	45	47	48	49	50
Co	ND	→	50	<500	ND	100	ND	100
Cr	100	ND	→	→	60	ND	→	→
Ni	40	→	→	<100	30	40	<100	40
Mg	<20	<10	→	<30	ND	15	<10	10
Rh	ND	→	→	<300	50	36	200	40
W	ND	0.3-1.0%	0.3%	ND	→	→	→	→
Pt	ND	→	→	<100	30	50	<100	40
Cu	ND	→	→	<?	5	→	<30	10
PLUTONIA								
Failure mode								
Predominant fragments	Small	→	Large	→	→	Small	Moderate	Small
Nature	Friable	→	Normal	→	→	Friable	Normal	Friable
Relative fines	Many	→	Few	→	→	Many	→	→
Grain diameter (μm)								
Pole	49	50	40-50	70-80	50-60	20-50	30-60	20-40
Equator	80	100	30-40	50-70	60-70	40-60	20-60	30-40
Middle	40	40-80	50-100	50-60	30-40	20-50	70-120	20-40
Pore Volumes ^h (%)								
Pole	14.7	21.3	35 (est)	18.5	13.9	Crushed	17.8	22.8
Equator	30.5	10.8	10.7	21.0	14.4	26.4	14.9	18.0
Middle	13.5	14-34	11.1	11.4	25.1	8.3-10.3	16.5	22.3
Substoichiometry	None	→	→	→	→	→	→	→
Fines analysis ⁱ								
Sieve fraction (μm)								
+6000	0.3900	0.6309	0.9924	0.8809	0.8948	0.6183	0.8109	0.5180
2000	0.3138	0.0946	0.0009	0.0391	0.0464	0.1106	0.0735	0.2126
841	0.1139	0.0231	0.0012	0.0215	0.0208	0.0889	0.0302	0.1223
420	0.0471	0.0091	0.0011	0.0081	0.0078	0.0320	0.0146	0.0410
177	0.0385	0.0105	0.0013	0.0107	0.0091	0.0371	0.0125	0.0300
125	0.0109	0.0040	0.0005	0.0048	0.0030	0.0129	0.0056	0.0101
74	0.0168	0.0069	0.0006	0.0074	0.0040	0.0270	0.0108	0.0142
44	0.0211	0.0083	0.0005	0.0120	0.0055	0.0363	0.0166	0.0189
30	0.0177	0.0060	0.0006	0.0078	0.0043	0.0207	0.0102	0.0163
20	0.0108	0.0027	0.0003	0.0037	0.0021	0.0097	0.0058	0.0086
10	0.0136	0.0030	0.0005	0.0026	0.0014	0.0057	0.0063	0.0047
-10	0.0058	0.0008	0.0002	0.0012	0.0006	0.0015	0.0029	0.0032
Subsieve fraction max. size (μm)								
10	0.002981	0.000578	0.000037	0.000599	0.000200	0.000626	0.000221	0.001603
9	0.000362	0.000032	0.000000	0.000000	0.000000	0.000182	0.000269	0.000292
8	0.000509	0.000000	0.000044	0.000102	0.000029	0.000224	0.000189	0.000205
7	0.000341	0.000061	0.000020	0.000068	0.000079	0.000172	0.000379	0.000275
6	0.000376	0.000019	0.000012	0.000129	0.000019	0.000162	0.000279	0.000173
5	0.000466	0.000022	0.000004	0.000150	0.000064	0.000094	0.000368	0.000200
4	0.000350	0.000028	0.000011	0.000051	0.000081	0.000048	0.000212	0.000218
3	0.000107	0.000011	0.000002	0.000011	0.000064	0.000018	0.000045	0.000038
2	0.000188	0.000010	0.000008	0.000054	0.000038	0.000008	0.000114	0.000127
1	0.000102	0.000013	0.000009	0.000054	0.000006	0.000004	0.000081	0.000075
0.5	0.000024	0.000004	0.000001	0.000017	0.000001	0.000001	0.000020	0.000021
<0.5	0.000005	0.000000	0.000000	0.000003	-----	-----	0.000002	0.000004

^aThe cracks are probably superficial. Penetration of the PICS was not confirmed by metallography.

^bThe defect was a continuation of a hoop stress failure.

^cIdentified only with the front (impact face) or back of the PICS, not with either male or female hemispheres.

^dA tensile rupture crossed the vent, but the vent seemed to be closed in a subsequent metallographic section.

^eThe vents were open to helium, but there was plutonia in the shield disk openings.

^fMicrohardness values were obtained with a 136° diamond indenter at 50-g load.

^gThe values are the maximum in either hemisphere.

^hPore volumes were determined by D. Rohr, LASL, from photomicrographs at 100X by Quantimet (Image Analyzing Computers, Ltd., Cambridge, England).

ⁱFuel from the impacted FSAs was sized by D. E. Garinger, LASL, according to standard sieve analyses. Subsieve particles were evaluated by extraction replica techniques.

VI. PHOTOGRAPHIC DOCUMENTATION

Fig. 18.

This radiograph of MHFT 45 is illustrative of those (also 47, 48, and 50) in which the fuel spheres were broken after vibration and shipping.

Fig. 19.

These radiographs of MHFT 40 illustrate the occasional damage to a fuel sphere during aging. Left: after vibration and shipping; three exposures did not reveal any fuel separation. Right: after aging, a peripheral displacement indicates that the sphere is broken.

Fig. 20.

MHFT 45 impact assembly after hard-surface impact at 85 m/s following hot reentry. Impact was opposite the GIS cap. The impact can deformed symmetrically. The GIS body and cap both delaminated, but were held tightly in the can.

Fig. 21.

MHFT 49 impact assembly after hard-surface impact at 87 m/s following cold reentry. Impact was again opposite the GIS cap. The impact can deformed symmetrically. Both body and cap of the GIS delaminated. The impact shell was loose in the can.

Fig. 22.

MHFT 50 impact assembly after side-on hard-surface impact at 86 m/s following hot reentry. The impact can deformed asymmetrically, the greatest distortion of all elements being near the GIS cap (left). Some foil radiation shield material is stuck to the side of the can.

Fig. 23.

Vapor-deposited plutonia inside a PICS is illustrated in this aged hemishell from MHFT 41. Some iridium transport also occurred, but its effect was apparent only in photomicrographs. The closure weld shield had been displaced about 60°.

Fig. 24.

Fingerprint cracking in Type III iridium from the impact face of MHFT 45. X 100. Again, there is a thin layer of vapor-deposited iridium on the inner surface.

Fig. 25.

The closure weld shield from MHFT 41. The microhardness values represent a 50-g load.

Fig. 26.

Typical Type WC-n iridium from MHFT 40. X 100. There is a slight vapor deposit of iridium on the inner surface. The microhardness traverse was made at 50-g load.

Fig. 27.

Type WE iridium from MHFT 49. Again, there is a thin layer of vapor-deposited iridium on the inner surface. X 100.

Fig. 28.

Weld microstructures contained mixed grain sizes, although there was none of the very pronounced columnar growth common to the early assemblies. These sections are from the closure weld in MHFT 45. Etched. X 50.

Fig. 29.
Vapor-deposited iridium reached a thickness of 50 μm inside MHFT 39. Intergranular fingerprint cracking also occurred in the Type WC material. X 100.

Fig. 30.
A tensile failure by intergranular cracking in the back of MHFT 50. X 100. Ductility is shown by the necking in the Type III material.

Fig. 31.

A cross section of a vent assembly from MHFT 41. X 10. This vent was not impacted. It was closed to helium by PuO_2 , a complex CaSiO compound, and sintering in the frit.

Fig. 33.

A section of a badly battered vent assembly from the impact face of MHFT 47. X 10. The vent seemed to be closed.

Fig. 32.

A section of the frit from MHFT 41 near the annular port in the shield disk. X 500. The vent was closed by sintering, PuO_2 (light gray), and a glassy CaSiO compound (dark gray) in interstices in the frit.

Fig. 34.

The vent shown in Fig. 33. X 100. No nonmetallic material was seen in the central cavity of any vent assembly.

Fig. 35.

A section of the vent assembly from the impact face of MHFT 50. Although the laser-drilled hole does not appear to penetrate the PICS, a metallic deposit of iridium, tungsten, and iron formed at the portal.

Fig. 36.

Vent from MHFT 48 partially blocked with iridium fragments, apparently debris from the drilling. X 200.

Fig. 37.

The vent assembly from MHFT 48 at the shield disk port. X 100. Plutonia had accumulated here to a thickness of 0.3 mm. The vent was closed.

Fig. 38.

Dark spots on the backs of PISAs from MHFT 45 (Fig. 8) and 47 (Fig. 11) were zones of reaction with an unidentified impurity. Shallow grain boundary cracking and penetration to about 1-16 μm occurred. X 500.

X 15.

X 100.

Etched, X 100.

Fig. 39.

Plutonia structure in a polar region of the fuel sphere from MHFT 47. The fuel density is 86.1% of theoretical, and the grains are 50-60 μm in diameter.

X 15.

X 100.

Etched, X 100.

Fig. 40.

Plutonia structure at the equator of MHFT 47. The fuel density is 85.6% of theoretical, and the grains are 60-70 μm in diameter.

X 15.

X 100.

Etched, X 160.

Fig. 41.

Plutonia structure in the middle of MHFT 47. The fuel density is 74.9% of theoretical, and the grains are 30-40 μm in diameter.

Pole.

Equator.

Middle.

Fig. 42.

Helium bubbles in the fuel from MHFT 45. Etched. X 500.

VII. DISCUSSION OF RESULTS

A. Normal Handling and Vibration

The FSAs received at LASL had been vibrated at Mound Laboratory to a level calculated to represent what they would have experienced in heat source assemblies during launching.³ As far as could be determined, the vibration had little effect on fuel sphere integrity. The spheres in the vibrated FSAs were all cracked; however, spheres that had not been vibrated (observed in impact tests other than the SST and SVT) were also cracked. It is concluded, therefore, that all FSAs in a heat source assembly will contain cracked fuel from the time of assembly.

It was not possible to separate the effects of transportation from those of vibration when the FSAs were received. Consequently, the SVT gave no data on the amount of fine plutonia generated in the vibration phase alone. Neither is relevant information available from other sources at LASL, because all fuels that have been sized were from FSAs that had been vibrated either individually or in a heat source assembly.

B. Reentry Heat Pulse

The effect of a reentry heat pulse on the fuel was not determined separately. In the SST, however, it was slight

C. Hard-Surface Impact

The iridium in all the impact specimens cracked. In two of the seven, however, containment breaching was not confirmed. The failures were essentially of three types, fuel push-throughs, fingerprint cracks, and hoop strain cracks. Fuel push-throughs were the most frequent, occurring in six of seven tests. These are tensile and bending fractures at protruding edges of fuel fragments that existed before impact or were formed during impact. Fingerprint cracks occurred in four tests. These annular crack patterns are complex systems of tension and shear accompanied by a rotation of the elements formed. They are related to the hardnesses of fuels and impact shells. Hoop strain cracks occurred in three tests. These are essentially tensile tears, but with a bending component, in the peripheries of impacted PISAs. They are

related to fuel friability and iridium's lack of ductility. All failures were intergranular, except some fingerprint cracks in which transgranular fracturing also took place.

There seems to be little choice among iridium sources, except, possibly, on the basis of ductility. All the materials (ORNL WE and WC, EI Type III) failed equally by fuel push-throughs and fingerprint cracking. None of the Type III shells failed from hoop strain. In one fuel push-through in a Type III shell, there was evidence of ductility, although the fracture mode was, again, intergranular. Improved iridium properties would obviously help,* although the defects were also related to fuel properties. Here only an optimum balance of hardness (density) and porosity would alleviate the situation. There were no detectable differences in the results from impacts on concrete and on granite targets.

VIII. SAFETY ASPECTS

Alpha contamination of the PISAs (residual or under decontamination shields) was confined within the impact shells. Any plutonium vapor that escaped through the vents during aging was also confined, because neither was the aging furnace contaminated nor was any detectable quantity of radioactive material spread from the FSAs before they were canned for impact. The level of contamination inside unimpacted GISs was determined, however, in only one instance when a total of 8 μg of plutonia was found in the graphite shell from MHFT 39.

Each post-test impact shell was analysed for total plutonium. The shells, together with cracked areas from the PICS they had contained, indicated that 400 g/mm² of plutonia might be expected to escape through a rupture produced on impact.⁵ Because damage to the GIS was always great enough to free the PISA were it not confined, the plutonia must be considered available for prompt release.

IX. STORAGE

The FSAs were held at a nominal 1210° C, three for 7.2 Ms, five for 2.6 Ms. These differences reflect three separate aging runs in which furnace capacity was limited to four units each (only three were available for the first run). The atmosphere to which all units were exposed during aging varied from

2.5×10^{-5} to 5.2×10^{-4} Pa. Fuel deterioration in four of the assemblies was evidenced in radiographs by displacement of fuel fragments relative to the main body, which indicated that the spheres were no longer intact. Sometimes further fragmentation appeared to have taken place. None of these conclusions is positive, however, because the orientation of the assemblies in the initial radiography was probably never duplicated, so previously undisclosed defects may have been revealed.

It is assumed that all vents were open and operative when the FSAs were received at LASL. After aging, at least one vent in all but one PISA was found to be open. After re-entry, impact, and defueling, both vents were found open in two units, one open in each of four units, and both closed in one. Vapor transport of material within the PISAs was the major cause of vent failure.

A complex glassy material, almost certainly vapor-deposited during aging, appeared to contribute most to vent clogging. Lowering the simulated operating temperature from 1268°C (in the SST) to 1210° did not alleviate the condition. Vapor transport of plutonia also contributed to clogging, but to a lesser extent. Vapor-deposited iridium present on the interior surfaces of the PICS did not appear to contribute to vent failure. The source of the impurity elements in the glass is not known. Although the major constituents correspond with impurities in the iridium, the plutonia seems a more likely source.

The significance of the glass in the vents at operating temperature is not clear. There has been no evidence that the PICS swelled during either the SST or SVT where the glass, seen during metallography of vents at room temperature, appeared to cause the clogging. Indeed, FSAs in current compatibility tests at comparable temperatures show intermittent helium release, as though the gas accumulates until enough pressure is generated to burst a liquid barrier.⁶ If this interpretation is true, vent clogging in the SVT should not have been a problem until some later date. Metallography of vent frits often showed a sintered structure that could account for, or contribute to, vent failure. However, because the frits are produced at a temperature about 700° C higher than those of the SVT, and because each assembly undergoes an acceptance test, such an interpretation seems unlikely.

There seemed to be no iridium degradation during the test. Extremely large iridium grains, equal to or larger in diameter than the wall thickness, were not uncommon. Although the stage in which they grew was not determined, preassembly heat treatment of the piece parts excludes aging from being more than

*ORNL has made an intensive effort to strengthen iridium by alloy addition.

a contributory factor. Although the large grains were deemed undesirable, intergranular fracturing occurred as frequently in regions of smaller grains.

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