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

by

E. M. Cramer

ABSTRACT

Five fueled-sphere assemblies similar to those proposed for use in the multihundred-watt radioisotope thermoelectric generator were subjected to conditions simulating earth impact after orbital abort. Of the five, one had no visible cracks, two had superficial cracks whose penetration of the iridium containment shell was not verified by metallography, one was obviously ruptured, and the fuel of one was exposed.

The basic causes of containment failure were as follows. (1) Large-grained iridium provided short, straight boundary paths susceptible to intergranular fracturing. (2) Large plutonia fragments produced excessive tensile strain in the containment where it was forced to bend over their projecting edges at the moment of impact. (3) Vents failed because of sintering and CVD of nonmetallic materials in the filter frits. (4) Of less significance, directional grain growth in closure welds apparently caused one failure.

1. INTRODUCTION

Because of the emergence of the modular multihundred-watt (MHW) radioisotope thermoelectric generator (RTG) as a power source for spacecraft, and because of increased concern over possible environmental contamination by radioactive material, a sequential test plan¹ for evaluating RTG containment capability was conceived. The plan was designed to simulate, with live fuel, an orbital phase abort resulting in atmospheric reentry of the generator, followed by earth impact and exposure of its components.

The MHW RTG heat source contains 24 individual fueled-sphere assemblies (FSAs). Each FSA is a $^{238}\text{PuO}_2$ sphere, about 37.21 mm (1.45-1.48 in.) in diameter, encapsulated in a vented postimpact

containment shell (PICS) of 0.51-mm (0.020-in.)-thick iridium* that is, in turn, enclosed in an 11.81-mm (0.465-in.)-thick graphite impact shell (GIS) of carbon-impregnated Thornel yarn.** The 24 FSAs are assembled into an aeroshell that provides basic protection for the heat source during reentry. The postreentry remains of the aeroshell and the GIS protect the individual FSAs during impact and leave the noble-metal PICS of each post-impact sphere assembly (PISA) to contain the plutonia until the debris can be recovered. For practical purposes, therefore, the FSA was selected for impact testing.

*This specification was superseded. The present dimension is 0.56-0.71 mm (0.022-0.028 in.).

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To serve the objectives of the safety program, the life of an RTG was divided into prelaunch, launch and ascent, and orbital phases. The Los Alamos Scientific Laboratory (LASL) was to evaluate the heat source response in the orbital phase only, to determine the response of FSAs subjected to approximations of their projected service history in an orbiting generator. The scope of the program is shown in Fig. 1.

In the course of the Sequential Safety Test (SST) program, seven FSAs were subjected to simulated launching vibration at Mound Laboratory and then to short-term aging at LASL. The aging was at RTG operating temperature and simulated its orbital lifetime. Of these FSAs, one was examined after aging, in launch abort simulation. Another was subjected to reentry only and then examined. Four were reentered and impacted under worst credible conditions, except for impact velocity. Two of these FSAs were then examined. Of the other two, one was buried in dry soil in air for 5.8 Ms (about

67 days) and the other was placed on a bed of soil and subjected to periodic wetting and drying for 31.54 Ms (1 yr). The seventh FSA served as a standby throughout these tests and then was impacted without reentry at the lowest credible impact temperature.

Planned long-term aging was deleted from the program. Preliminary USAF and AFWL calculations had shown a relatively high probability of orbital reentries, and the AEC Space Nuclear Systems (SNS) Division suggested that both the SST and the subsequent Safety Verification Test (SVT) be used to provide performance data in the most probable situations.²

The test parameters, specified by the prime contractor, General Electric Co.,² were as follows. Vibration: Acceptance level at 996°C.

Aging: 1268°C (GIS) for 30 days. (One-dimensional equivalent of the outer surface of the GIS.)

Reentry profile: Stagnation line FSA at minimum gamma conditions. (See Fig. 2.)*

Impact velocity: 74.7-78.0 m/s (245-256 fps).

II. PROCEDURES

Implementation of the test plan shown in Fig. 1 included the following operations.

*Figure 2 shows the GIS temperature profile in the LASL gun calculated to give a PICS temperature equal to the average profile of all the PICS in a generator.

Fig. 1. SST plan for MHW FSAs.

Fig. 2. SST impact shell reentry profile.

All FSAs were subjected to procedures A-C and E unless urgent need for data precluded completion of the scheduled inspections, as happened in several instances. Four FSAs were subjected to procedure D according to the test plan.

A. Pretest inspection

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

B. Short-term aging

1. Heat treat the FSAs at a nominal 1268°C, measured optically on the surface of the impact sphere, for 2.6 Ms (30 days) in a vacuum.

C. Post-aging inspection

1. Perform radiography.
2. Weigh and measure the FSA.
3. Remove, weigh, and measure the PISA.*
4. Perform vent check by helium release during self-heating.
5. Reassemble FSA.

D. Impact test

1. Weld FSA into impact can.
2. Subject FSA to reentry thermal pulse.
3. Impact on a granite target.
4. After impact, bury one assembly in dry soil. Place another on soil and expose it to simulated rain, wind, and sun.

E. Postimpact examination and disassembly

1. Recover the PISA from the impact debris.
2. Inspect visually for ruptures and take documentary photographs.
3. Disassemble.
 - a. Defuel the postimpact containment shell.
 - b. Examine the PICS metallographically.
 - c. Examine the plutonia metallographically.
 - d. Perform plutonia fines analysis.
 - e. Take documentary photographs of significant effects.

*To facilitate the vent check, it was agreed that the PISA would be removed from the impact shell.

III. TEST FACILITY

The existing protective enclosures and metallographic equipment for plutonium handling were used in the posttest examinations.* The radiographic inspections and welding also were done in existing Laboratory facilities, most of which have been described in the literature.³⁻⁷ The welding facilities are described briefly in the section on impact testing.

Special equipment was provided for FSA aging, impact testing, and disassembly. These items and their application to the test program are described briefly in the sections on those subjects.

IV. AGING

The thermal experience of an FSA during orbital abort was simulated by a heat treatment at 1268°C, measured on the GIS surface, for 2.6 Ms (30 days). A carbon resistor furnace** with essential power supply and control instrumentation was installed for the purpose. This apparatus is shown in Fig. 3. The furnace chamber communicated with an inert-atmosphere glovebox so that the FSAs

*C. Land and L. Bergamo assisted in metallography, D. Rohr in image analysis, and D. Garinger in plutonia preparation.

**CENTORR Model 10-4X20G4-20 High-Temperature Furnace, CENTORR Associates, Suncook, NH.

Fig. 3. Facility for aging FSAs in a high vacuum and for storing assemblies.

could be charged without exposure to air. The glove-box also served as a place in which to open shipping containers and store assemblies. Four FSAs were aged at a time. During the aging, the pressure in the furnace chamber approximated 1.3×10^{-4} Pa (1×10^{-6} torr).

V. IMPACT TESTING

The ground impact of a reentering FSA was simulated by using a gas gun to fire it against a granite target at 74.7-78.0 m/s (245-256 fps). A vertical gas gun with an impact chamber, residual-energy absorbing equipment, gas supply, and the necessary instrumentation provided these conditions.⁸ Features of the gun tube and the projectile are shown in Figs. 4-6.

The test specimen, shown in Fig. 6, was enclosed in a tantalum container to keep the gun from being contaminated with plutonia. The 0.75-mm (0.030-in.)-thick tantalum container, which

Fig. 4. The gas gun breech assembly.

Fig. 5. The muzzle end of the gun.

also served as a susceptor to heat the specimen electronically during the reentry pulse, was closed by welding with an inert tungsten arc. (The welding was done in an argon-filled enclosure, but the gas was evacuated from the impact can before the test.)

A catch tube inside the impact chamber of the gun received the canned specimen. The catch tube became a sealed container as part of the projectile was driven into its tapered port, and it was later detached from the gun to be used in transporting the test body to the next station for disassembly. The impact can velocity was monitored by means of an array of shorting pins in the gun muzzle. The specimen temperature was evaluated by a thermocouple in contact with the impact can.

Fig. 6. The projectile assembly.

VI. FINES FORMATION DURING IMPACT TESTS

The plutonia fuel from the five impacted FSAs was sized according to standard sieve analysis. Sub-sieve particles were evaluated by extraction replica techniques. The analyses are listed in Table V in Sec. VII. Within the test context, the size distributions represent the following stages in the life of and FSA: 1. Prelaunch, including transportation, storage, vibration, and aging (FSA 28). 2. Prelaunch plus reentry (FSA 25). 3. Orbital reentry plus highest temperature hard-surface impact (FSAs 26 and 30). 4. Orbital reentry plus lowest temperature hard-surface impact (FSA 29).

*So far, heat source increments have been called FSAs. FSAs, however, include both multihundred-watt fueled test specimens (MHFTs) and multihundred-watt fueled specimens (MHFs) that were installed in generators rather than used in tests. To avoid confusion, the units will continue to be called FSAs. Note, however, that all of the FSAs described herein are test specimens.

Note that the tests involved only single FSAs. Therefore, the results of the tests that did not include impact can be applied to the FSAs in a heat source. They may not, however, be so applied when impact is involved, because the degree of fuel disintegration is a function of containment deformation which varies with location within the heat source.⁹

VII. POSTTEST EXAMINATION

A. FSA 28.

FSA 28 was withdrawn from the test after short-term aging, in accordance with the test plan in Fig. 1. As-received radiography indicated that the plutonia had been fractured badly by vibration and shipping and that the fuel sphere was broken. The assembly was not radiographed after aging.

The GIS exterior seemed unaffected by aging. Internally, a small pit with a white halo was found in a location corresponding to one of the vents. The PISA was clean and bright, except that one vent had a black incrustation of iridium with some tungsten and oxygen.

The PICS interior was heavily coated with chemical-vapor deposited (CVD) plutonia. Both vents were closed to helium by the effects of sintering in the frit and deposition of plutonia and a second nonmetallic material containing mostly calcium, silicon, and oxygen, also in the frit. However, no swelling of the PICS was detected.

The iridium grains were of mixed sizes, the diameter of the largest being equal to the thickness of the PICS wall. There were also large grains in the closure weld. Both of these conditions provided short boundary paths, susceptible to fracturing, across the PICS wall. However, no mechanical defects were seen in the shell.

The plutonia had fractured into large pieces with relatively few fines. Its grains were ~ 50 μm in diameter, and the pore volumes at the pole, equator, and middle were 17, 11, and 23%, respectively.

B. FSA 25.

FSA 25 was examined after vibration, aging, and reentry. Initial radiography had indicated that the plutonia was badly cracked and the sphere broken after vibration and shipping. After aging, the GIS seemed unaffected, and the fuel sphere had deteriorated further. A vent check was positive (at least one vent was open), and the FSA was reentered.

When the GIS was opened and the PISA removed, a crack about 2 mm long was found in the closure weld. Both vents were later found to be closed to helium, so it is presumed that the positive vent check was really a result of the fracture.

After testing, the PICS interior was heavily coated with vapor-deposited plutonia and some iridium. The iridium grain size approximated the wall thickness, and straight-through grain boundaries were characteristic both there and in the weld. The fracture mentioned above was intergranular and had progressed through such a boundary. The iridium microhardness and impurity content were normal, except for the silicon content which was slightly high. The vents were closed by the effects of sintering and CVD plutonia in the frit.

The plutonia had fractured into many medium-size pieces. Its grains were 40-80 μm in diameter. Pore volumes at the pole, equator, and middle were 19, 10, and 17%, respectively.

C. FSA 26.

FSA 26 was the first FSA to be impacted. Initial radiography had indicated that the plutonia was not an intact sphere after vibration and shipping. The FSA was not radiographed after aging. After aging, the GIS exterior seemed unaffected. There was a small pit with a white halo on the inside of the cap. The PISA, including the vents, was clean and bright, and a vent check was positive.

Accidental impact on the edge of the cap crushed the GIS badly and deformed the PISA asymmetrically. There were no visible ruptures in the PICS, but at least one fingerprint-type crack in the impact face penetrated the containment wall.

The fracture was intergranular. Maximum iridium grain size again approximated wall thickness in both hemishells and in the closure weld. The vents were closed to helium after impact by the effects of sintering and CVD plutonia in the frits.

The plutonia had deformed by plastic flow and was broken into many medium-size pieces. Its grain diameters were 40 to 50 μm , and pore volumes at the pole, equator, and middle were 11, 12, and 35%, respectively.

D. FSA 27.

FSA 27 was the second FSA impacted and the first to be environmentally exposed. Initial radiography had indicated that the plutonia was not an intact sphere after vibration and shipping. The FSA was not radiographed again after aging.

After aging, the GIS was clean and bright. Internally, a small pit with a white halo occurred in the cap, presumably at a vent site. The PISA, including both vents, was also clean and bright. A vent check by helium release was positive.

Impact was 180° from the GIS cap, as intended. The GIS body was not split, but, in addition to having a crushed cone of material in the impact face, it delaminated when removed from the impact can. The PISA was evenly deformed. There appeared to be a small crack about 3 mm long in a fold in the impact face at the edge of a plutonia fragment. Shell penetration, however, had not been verified when the PISA was placed in the environmental chamber.

The PISA was recovered for examination after 1-yr exposure to periodic rain and drying.¹⁰ During the first rain, it sank into the soil to about nine tenths of its height, probably because of cavitation in the loose soil. As the soil became compacted, settling ceased.

The exposed PISA surface was covered by a scale deposited as the rain evaporated. The buried part had a thick coating of loosely bonded soil particles. The soil had not fused. Aside from the superficial coatings, the PICS was unaffected by exposure.

E. FSA 30.

FSA 30 was the third FSA impacted. Initial radiography had indicated that the plutonia was no longer an intact sphere after vibration and shipping. However, no further deterioration was seen during aging.

Impact was 180° from the GIS cap. The graphite body was not split, but a cone of material in the impact face was crushed and there was some delamination of the cap. The PISA was evenly deformed. A fingerprint pattern of short cracks occurred in the impact face, but metallography did not verify penetration of the iridium shell.

At disassembly, the PICS interior was heavily coated with CVD plutonia. A vent in the impact face was closed to helium by a combination of sintering* and CVD plutonia, plus a second nonmetallic phase in the frit which contained mostly calcium, silicon, and oxygen. Iridium impurities were at a normal level.

The plutonia had fractured mostly into relatively small fragments. Grain diameters varied from 40 to 80 μm at the equator to the original particle size at a pole and at the middle. Pore volumes at the pole, equator, and middle were 14, 4, and 16%, respectively. The plutonia appeared to have been substoichiometric with respect to oxygen at some time during its thermal history, thereby allowing partitioning to occur. (See Fig. 34, Sec. VIII.) Its oxygen:metal ratio, determined by thermogravimetric analysis, was 1.90.

F. FSA 31.

FSA 31 was the fourth FSA impacted and the second environmentally exposed. Initial radiography had indicated that the plutonia was badly cracked but still intact. No further deterioration was apparent after aging.

After aging, the GIS was clean and bright. Its interior was dull but had no detectable pits. The

*Sintering is considered to be a factor in vent failure. It was not established, however, whether the varying degree of sintering observed occurred when the filter was made or during aging, for example.

PISA, including both vents, was also clean and bright. A vent check by helium release was positive.

Impact was 180° from the GIS cap. The graphite body was split. Its cap had been crushed and delaminated. The PISA deformed evenly, although the effect of large fuel fragments was visible.

Tensile ruptures about 2 mm long occurred in the PICS periphery and along a reentrant fold in the impact face at the edges of large fuel fragments. The PISA was buried 0.229 m (9 in.) deep in dry soil for 5.8 Ms (67 days) before further examination.

Surface thermocouples showed a PICS temperature of 1135-1150°C during the environmental test. When recovered, the PISA had a thick coating of fused soil particles. Where the fused material was continuous and in intimate contact with the iridium, no erosion took place. The soil coating, however, was fissured, and there the iridium was so eroded that the wall was often perforated. Analysis of residual nonmetallic material in the erosion sites showed it to be an oxide of iridium, and the erosion mechanisms to be oxidation and vapor transport. There was no evidence of reaction between soil and iridium.

Chemical analysis of the fused soil showed that it contained 0.03 wt% plutonium. Assuming a shell thickness of 5 mm and a sintered density of 2, the weight of plutonia released was 0.02 g.

G. FSA 29.

FSA 29 had been held as a replacement in case one of the first six assemblies was accidentally destroyed. With completion of the six tests, SNS agreed that it be impacted at the lowest credible temperature rather than used to supplement a preceding test.

Initial radiography had indicated that the plutonia was no longer an intact sphere. After aging, the impact shell seemed unaffected both internally and externally. The PISA, including both vents, was clean and bright. A vent check by helium release was positive.

Impact was 180° from the GIS cap at 1093°C without a reentry pulse. The GIS cap had been crushed by the impact can, but the body was not split. The PISA deformed unevenly over large fragments of plutonia. The PICS was ruptured in all surfaces. Six hoop stress cracks occurred in the periphery. Several tears occurred over fuel fragments in both the back and the impact face. A fingerprint pattern of cracks also appeared in the impact face. Both vents were open to helium.

Impurity content, microhardness, and grain size in the iridium were normal.

The plutonia had fractured into small pieces. Its grain size varied from 50 to 80 μm , and pore volumes at the pole, equator, and middle were 16, 12, 15%, respectively.

The information presented in this section is included in Tables I through V, but the tables are more complete and allow easier comparison of individual samples.

TABLE I
TEST HARDWARE AND PRE-LASL HISTORY

	FSA 25	26	27	28	29	30	31
FUEL SPHERE	SPO-50	51	52	53	57	58	60
Oxide weight (g)	253.8	253.9	253.8	253.8	253.0	253.1	253.8
Batch number	P286	P287	P289	P291	P294	P301	
Plutonium weight (g)	221.4	222.0	219.1	221.1	220.0	220.5	
Pressing date	3/20/73	4/09/73	4/11/73	4/11/73	4/25/73	4/26/73	5/10/73
Density (% theoret)	82.16	82.46	82.72	82.57	81.63	81.93	82.69
Outgassing time (h)	1						
temperature (°C)	1500						
Diameter (mm) pole	36.98	36.83	36.91	36.86	37.06	36.91	
equator	37.44	37.47	37.31	37.34	37.39	37.19	37.31
CONTAINMENT SHELL							
Male	S/N 846-51	850-51	841-50	837-51	849-51	848-51	695-44
Coated disk	93 Lot 1	100 Lot 1	116 Lot 1	125 Lot 1	135 Lot 1	133 Lot 1	--- Lot 1
Shield disk	67	33	71	73	79	77	61
Decon disk	55	57	58	61	70	67	72
Weld shield	24	25	26	27	30	29	31
Laser hole size (in.)	0.0044/45	0.0055/47	0.0049/50	0.0050/40	0.0045/43	---	---
Female	S/N 827-51	830-50	831-50	835-51	821-51	853-52	590-44
Coated disk	94 Lot 1	112 Lot 1	120 Lot 1	126 Lot 1	99 Lot 1	134 Lot 1	--- Lot 1
Shield disk	68	70	72	74	80	78	82
Decon disk	56	58	60	62	71	69	73
Laser hole size (in.)	0.0046/47	0.0046/44	0.0046/43	0.0047/44	0.0045/44	---	---
Preassembly heat treat.	1500°C, 1 h						
Coated disk heat treat.	1000°C, 1 h						
Welding date	4/18/73		5/01/73				5/24/73
IMPACT SHELL							
Body	S/N 861561	861563	861574	861575	861578	868127	868129
Cap	S/N 861520-1	861523-1	861521-2	861516-1	861517-1	868157-2	858158-1
Type	Threaded						
Assembly date	4/23/73		5/01/73				5/24/73
Vibration test	1000°C						
Heat treatment	None						
Radiography	PuO ₂ cracked but intact.				PuO ₂ cracked and broken	Indeterminate.	No defects detected.
Shipped to LASL	6/13/73				6/20/73	7/03/73	

TABLE II

PRETEST INSPECTION

	FSA 25	26	27	28	29	30	31
Radiography	PuO ₂ badly cracked						Intact
	Sphere broken						
Diameter (mm) pole	65.38	Cap flush				64.82	64.87
equator	64.97	64.77	64.82	64.85	64.90	64.85	64.90
Weight (g)	446.1	446.5	446.4	445.3	445.9	443.7	445.7
Appearance	FSA clean and bright						

TABLE III

SHORT-TERM AGING

	FSA 25	26	27	28	29	30	31
Time (ms)	3.0			2.7	2.6		
Temperature (°C)	1272				1265		
Appearance	No effect noted						
Radiography	PuO ₂ badly cracked						
	Sphere broken						
Vent check	Open			Closed	Ir distorted	Sphere broken	
Diameter (mm) pole	Not taken			64.72	Open	Closed	Open
equator	Not taken			64.80	64.85	64.82	64.85
Weight (g)	445.2	Not taken	446.1	445.2	444.8	443.7	445.6
Diameter (mm) pole (PISA)	40.10	Not taken	40.13	40.41	39.70	40.06	40.44
45°	40.61	Not taken	40.61	40.69	40.56	40.61	40.69
135°	Not taken				40.67	40.54	40.72
weld	Not taken		41.00	41.14	Not taken		
Alpha count (1000 cpm)							
body cav (GIS)	Not taken	> 100	Not taken	> 100	Not taken		
cap cav	Not taken			> 100	Not taken		
surface	~0.0250	Not taken		~ 3	~0.150	~0.100	~ 1
	(swipe)				(swipe)	(swipe)	(swipe)
Visual inspection (PISA)	Rupture in weld	PISA clean and bright			Vents clean		
	Vents clean	Vents clean			Vents clean		
	1 vent clean	Pit in GIS		1 vent clean			
	1 gray dep			1 black dep			
				Pit in GIS			

* Later visual inspection revealed a rupture in the weld which probably accounts for this open vent result.

TABLE IV

IMPACT TEST

	FSA 25	26	27	29	30	31
Date		8/27/73	8/29/73	1/11/74	11/13/73	11/15/73
Vent check	Not taken			Open	Not taken	
Reentry pulse	Standard			No reentry	Standard	
Radiography	Some damage to PuO ₂	---	---	---	---	---
Diameter (mm) pole (PISA)	40.21	---	---	---	---	---
45°	40.72	---	---	---	---	---
135°	40.72	---	---	---	---	---
On weld	41.04	---	---	---	---	---
Velocity (m/s)	---	75.29	75.89	76.2	76.20	74.07
Temperature (°C)	---	1550	1550	1093	1550	1550
Target	---	Granite				
Orientation	---	60° from cap*	180° from cap			

*The impact can was changed during welding. The FSA apparently was replaced incorrectly.

TABLE V

POSTTEST EXAMINATION

	FSA 25	26	27	28	29	30	31
IMPACT CAN							
Deformation	---	Assymetrical	Symmetrical	---	Symmetrical		
Failure	---	Intact		---	Intact		Punctured
IMPACT SHELL (GIS)							
Failure	---	No longer coherent	Body not split Delaminated	---	Body not split Cap crushed	Cap, body intact except impact face	Body split Cap crushed
Impact face		In place					
Alpha count (1000 cpm)							
body cavity	>100				Not taken	~ 80	> 100
cap cavity	>100				Not taken	~ 80	> 100
surface	~ 50	~ 20	> 100	~ 3	Not taken	> 100	~ 85
CONTAINMENT SHELL (PICS)							
Deformation	---	Very irregular	Regular except fold in IF	---	Irregular	Regular except fold in IF	
Diameter (mm) maximum	---	42.94	42.62	---	43.64	42.75	42.82
minimum	---	42.01	42.11	---	42.42	42.01	41.58
Height (mm)	---	30.76	37.06	---	30.00/ 31.57	30.76	31.17
Failure							
Tensile cracks-back	0				4	0	
Length/width (mm)					2-3/0.5		
Tensile cracks-hoop	0				6	0	2
Length/width (mm)					1-12/0.1-1		2/0.5
Tensile cracks-impact face	0		1	0	4	0	3
Length/width (mm)			3/0.5		1-5/0.5		2/0.5
Fingerprint cracks-IF	u	Many	0		1/2 of IF	Many	0
Length/width (mm)		1-5/0.5			1-3/0.2	1/0.1	
Closure weld (mm)	2/0.02	0					
Type of fracture							
Tensile	---	---	Apparent intergran.	---	Intergranular	---	Apparent intergran.
Hoop	---	---	---	---	IG	---	---
Fingerprint	---	IG+TG	---	---	IG	IG	---
Weld	IG	---	---	---	---	---	---
Interior of PICS							
CVD iridium	Yes		No	Possibly	Yes	No	Possibly
Thickness (μ m)	<10			<5	10-20		<5
CVD PuO ₂	Yes	No	Yes				
Thickness (μ m)	<20	Indeterminate	Loose				
General stain	No	Yes	No		Yes		
Reaction with PuO ₂	None						
Bubble test of vents							
Impact face	Closed				Open	Closed	
Back	Closed				Open	V sl open	Eroded away
Cause of blockage							
Sintered Ir in frit							
CVD PuO ₂					PuO ₂ present	CVD PuO ₂	
Ca/Si/O					Ca/Si/O ^d	Ca/Si/O	
Typical microhardness ^c (VHN)							
Impact face	Not taken	310	400	Not taken	370	360	320
Back	335 (2 hemi)	310	340	350 (2 hemi)	365	345	325
Closure weld	330	315	340	350	365	350	Not taken
Weld shield	Not taken	340	Not taken		390	370	Not taken
Grain diameter (μ m)	170 to 508 or 1 to 3 grains across the 0.020-in wall thickness.						
Selected impurities ^e							
Al (ppm)	50/50	10-100/10/100	30/<30	30/<20	30/30	40/40	^b
Si	100/100	10-100/10-100	15/15	40/10	10/30	50/50	
Fe	100-1000/ 100-1000	100-1000/ 300-3000	200/300	200/200	400/800	400/400	
Mo	<300/<300	<100/<100	300/300	<300/<300			

TABLE V. (Continued)

	FSA 25	26	27	28	29	30	31
							b
Cr	<30/<30	10-100/ 10/100	30/50	20/20	200/300	50/100	
Ni	50/50	10-100/ 10/100	30/30	50/50	60/100	100/50	
Co	50/50	<100/<100	100/<100	50/50	60/150	100/100	
PLUTONIA							
Failure	Fragmentation, moderate size pieces	Plastic flow and fragment Moderate size	Fragmentation Moderate fines	Fragmentation Mostly lge Few fines	Fragmentation Mostly small		
Grain diam (μm) ^f							
Pole	40-80	40-60	50-80	40-60	40-80	60-80	
Equator	60-80	40-60	50-80	40-60	40-80	Particle size	
Middle	60	40-60	50-80	40-60	Particle size	Particle size	
Pore volumes (%) ^g							
Pole	18.7	11.5	18.4	17.3	6.6	14.3	
Equator	10.2	11.2	11.9	10.5	14.4	4.3	
Middle	17.3	34.9	14.7	23.1	14.3	16.1	
Oxygen/metal ratio ^h	Not determined				1.98	1.99	
X-ray diffraction pattern ⁱ	Not determined				PuO _{2-x}	PuO _{2-x}	
Fines analyses (μm)							
Sieve fraction +6000	0.5669	0.6946	0.8727	0.9647	0.4655	0.7547	
+2000	0.0228	0.1414	0.0716	0.0244	0.2671	0.1939	
+ 841	0.0039	0.0732	0.0244	0.0058	0.1443	0.0171	
+ 420	0.0019	0.0244	0.0083	0.0021	0.0405	0.0060	
+ 177	0.0014	0.0183	0.0064	0.0013	0.0326	0.0082	
+ 125	0.0004	0.0064	0.0021	0.0004	0.0087	0.0047	
+ 74	0.0005	0.0130	0.0035	0.0004	0.0109	0.0059	
+ 44	0.0005	0.0136	0.0044	0.0003	0.0084	0.0042	
+ 30	0.0003	0.0033	0.0032	0.0001	0.0084	0.0023	
+ 20	0.0002	0.0080	0.0019	0.0001	0.0053	0.0010	
+ 10	0.0005	0.0031	0.0011	0.0002	0.0065	0.0013	
- 10	0.0007	0.0008	0.0006	0.0002	0.0017	0.0007	
Subsieve fraction(μm)							
10	0.000423	0.000435	0.000001	0.000135	0.000805	0.000128	
9	0.000095	0.000029	0.000002	0.000009	0.000117	0.000187	
8	0.000050	0.000040	0.000008	0.000006	0.000082	0.000000	
7	0.000045	0.000095	0.000021	0.000016	0.000110	0.000000	
6	0.000061	0.000085	0.000021	0.000006	0.000174	0.000083	
5	0.000041	0.000035	0.000033	0.000007	0.000080	0.000112	
4	0.000028	0.000058	0.000028	0.000005	0.000103	0.000082	
3	0.000018	0.000030	0.000000	0.000003	0.000123	0.000049	
2	0.000009	0.000024	0.000034	0.000003	0.000055	0.000041	
1	0.000002	0.000006	0.000072	0.000001	0.000015	0.000012	
0.5	0.000000	0.000002	0.000329	0.000000	0.000004	0.000004	

a FSA 27 was subjected to a 1-yr postimpact environmental test.

b FSA 31 was buried in soil for 67 days after impact and was not analyzed for impurities and grain size.

c Numbers are 136° diamond pyramid hardness values at 50-g load.

d Electron microprobe analyses show that this material contains mostly calcium, silicon, and oxygen with magnesium or aluminum and, sometimes, plutonium.

e The analyses are from both hemispheres. Impurities selected are those having the greatest concentration or showing the greatest variation.

f The values are approximations of actual grain size, not from an intercept, so they are based on the large grains unless more than one range is evident.

g Pore volumes were determined from photomicrographs at 100 X by Quantimet (Image Analyzing Computer, Ltd., Cambridge, England).

h Thermogravimetric analyses were made after storing the plutonia in air for several days.

i X-ray diffraction analyses were made after storage in air.

VIII. PHOTOGRAPHIC DOCUMENTATION

Effects observed during the test series were photographed whenever practicable. However, because of the similarity of the specimens and test conditions, the effects differed mostly in degree. Consequently, only a typical example at each stage of the test is included here, except where a unique result warrants attention. Each example is identified by FSA number so that the effect can be related to the specific material and test conditions that produced it.

The metallographic iridium specimens were mechanically polished with alpha alumina on nylon cloth in a vibratory polisher and finished by hand with 1- μ m diamond on a high-napped synthetic cloth. The polished surfaces were electrolytically etched

in a solution of 20% hydrochloric acid and 80% water saturated with NaCl at 7.5 volts ac. The metallographic plutonia specimens were pressure potted in epoxy resin, ground through 600-grit silicon carbide paper, and mechanically polished with alpha alumina on nylon cloth in a vibratory polisher. The polished surfaces were chemically etched in a solution of 10 ml of hydriodic acid, 10 ml of hydrochloric acid, and about 1 ml each of nitric and hydrofluoric acids.¹¹

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

Fig. 7. Radiographs of FSA 25 at 0 and 90° before shipment. The plutonia is cracked, and the sphere seems to be broken. (Copies of Mound Research Corporation photographs 730954 and 730955.)

Fig. 8. Radiographs of FSA 25 through the pole (relative to the primary container) and equatorially at 0 and 90° after shipment. The fuel sphere seems to be broken.

Fig. 9. Radiographs of FSA 25 at 0, 120, and 240° after short-term aging. The plutonia sphere is broken.

Fig. 10. Radiographs of FSA 25 at 0, 120, and 240° after aging and a reentry heat pulse. The reentry pulse has damaged the plutonia sphere further.

Fig. 11. FSA 25 after aging. None of the assemblies seemed to be affected by the heat treatment.

Fig. 12. The interior of the FSA 28 GIS. Pitting in the FSA 26 and 28 shells is assumed to be at the location of a vent in the PICS.

Fig. 13. The impact assembly containing FSA 30. The tantalum impact cans deformed symmetrically except when impact was on the threaded joint between the GIS cap and body.

Fig. 14. FSA 30 in place in half of its impact can. The FSA was loose in the can. Its cap was delaminated, but the GIS body was not split. Crushed material in the impact face is still in place between the PISA and the can.

Fig. 15. Half of the FSA 29 GIS in place. The shell cap was delaminated, and the body was split. Crushed material in the impact face is still in place.

Fig. 16. A profile of the FSA 29 PISA. Note the tensile hoop rupture in the periphery of the flattened sphere.

Fig. 17. A fingerprint pattern of cracks occurred in the impact face of the FSA 26 PISA. Distortion is greater than normal because impact was on the threaded joint in the GIS.

Fig. 18. Tensile tearing over the edges of large plutonia fragments occurred in the FSA 29 PICS. Although the defects are not visible, the extent of deformation at such locations is clear.

Fig. 19. The FSA 31 PISA after burial in dry soil for 5.8 Ms (67 days). A thick layer of fused soil particles formed over its surface. Where the fused coating was fissured, extensive iridium erosion by oxidation and vapor transport occurred. (See also Figs. 29 and 30.)

Fig. 20. The FSA 27 PISA after exposure to periodic rain and drying on a soil bed in an environmental chamber. Except for discoloration and the superficial scale deposit, the PICS was not affected by exposure.

Fig. 21. A typical vent section from FSA 25. X 10. Although undamaged by impact, it was closed by the effects of sintering and vapor-deposited plutonia in the frit. A clip to support the section and an alligator clip for electrical contact appear in cross section above and below the vent assembly.

Fig. 22. Sintering and vapor deposition of plutonia in a frit is shown in this section of FSA 26. X 500. Sintering occurred generally throughout the frit, whereas the plutonia was deposited mostly between the shield and fritted disk near the hole in the shield.

Fig. 23. A dark nonmetallic material containing calcium, silicon, oxygen, and magnesium or aluminum also contributed to vent failure in FSA 31. X 500. It occurred less frequently than plutonia (light areas) but in the same locations and always associated with the oxide.

Fig. 24. A dark incrustation surrounded one vent in FSA 28. The material is shown in cross section in Fig. 25.

Fig. 25. A section through the portal of the vent shown in Fig. 24. X 250. The incrustation contained iridium, tungsten, and oxygen. Condensed and solidified iridium lines the vent hole.

Fig. 26. A grain boundary fracture through the closure weld in FSA 25. X 100. The fracture was detected after aging, but neither the time nor the cause of its occurrence is known.

Fig. 27. Fingerprint cracks were essentially of the grain boundary type. These, in the impact face of FSA 26, ruptured the containment wall. X 100.

Fig. 28. Tensile hoop and push-through ruptures were also intergranular. This push-through, at the edge of a plutonia fragment in FSA 26, illustrates the nature of these failures. X 100.

Fig. 29. Erosion of the FSA 31 PICS wall from burial in dry soil. X 100. The containment wall was perforated in several places. Figure 30 shows further details.

Fig. 30. Part of the eroded containment wall shown in Fig. 29. X 100. Residual nonmetallic material in the spongy iridium contained only iridium and oxygen, indicating that erosion was by oxidation and vapor transport. There was no evidence of impurity diffusion or grain boundary attack.

Fig. 31. Plutonia from the FSA 25 fuel sphere, exclusive of fine material. These fragments show the condition of a fuel charge after reentry heat pulse and before impact.

Fig. 32. Plutonia structure in the polar, equatorial, and middle regions of the FSA 25 fuel sphere. X 15. Typically, density in the equatorial region is greatest.

Fig. 33. Plutonia microstructure in the middle of FSA 25. X 100. The porosity was 17.3 vol %, and the grain diameter was about 60 μm .

Fig. 34. Plutonia microstructure in the middle of FSA 30. X 100. The material appears to have an oxygen:metal ratio of 1.85, but the actual ratio was 1.99. It is presumed to have been deficient in oxygen at some time during its thermal history when partitioning took place; then it was oxidized to PuO_2 but retained a metastable ghost of its former state.

IX. RESULTS

The initial damage that affected the SST outcome occurred early in the history of each FSA. Radiography at Mound Laboratory revealed severe plutonia cracking after the vibration phase, although it may have occurred even earlier. Large fragments of fuel present during impact strained the containment shell of two, or possibly three, units to failure where the material stretched over their projecting edges. The fuel disintegrated further in subsequent thermal treatments, but this did not appear to affect the outcome.

Grain growth in the iridium often progressed to the extent that the shell wall was only one grain thick. Because the failures seen in the iridium were essentially intergranular, these short, direct, boundary paths increased the susceptibility to fracturing. However, this defect was serious in only one instance. As it would have entailed destructive testing, we did not determine the phase in which the growth occurred. Probably all three high-temperature phases, welding, heat treatment, and aging contributed. Evidently, as it was not confined to heat-affected zones, the growth did not occur during welding alone.

Vent failure usually resulted from a combination of iridium frit sintering, transport of plutonia to the frit, and the presence in the frit of a nonmetallic material that contained calcium, silicon, and oxygen and, sometimes, plutonium, iron, and magnesium or aluminum. We do not know the source of this material. Its major constituents are impurities normally found in the iridium, but the plutonia seems a more probable source. Vapor transport of iridium from the weld shields to the PICS seemed not to affect the vents. There was no evidence that the thermal treatments degraded the iridium by reaction with either plutonia or graphite.

Plutonia inhomogeneity was another bad condition, but, again, specific effects cannot be ascribed to it. Perhaps it contributed to iridium

failure by influencing the size of fragments present at impact, or contributed to formation of finger-print cracking in the impact face. This crack pattern results from hard fuel, but the location of the densest zone (usually the equatorial zone) relative to the impact face is not known.

The primary objective of the test series was to evaluate the capability of the hardware to contain plutonia, which it did very well. No significant amount of plutonia escaped into the aging furnace. After aging, the junction of the impact sphere cap and body gave an alpha swipe count of about 200/min. Some internal alpha counts exceeded 100 000/min, but the contamination was essentially confined within the GIS. The SVT* indicates that release of milligram quantities of plutonia from impacted assemblies certainly would have occurred in one instance (FSA 29) and in two instances (FSA 29 and 31) at most.

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*The SVT succeeds the SST and is similar except that the hardware is flight qualified. A report on the SVT is in preparation.

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