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***General-Purpose Heat Source Development:
Safety Verification Test Program***

Bullet/Fragment Test Series

MASTER

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Los Alamos, New Mexico 87545

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GENERAL-PURPOSE HEAT SOURCE DEVELOPMENT: SAFETY VERIFICATION TEST PROGRAM

Bullet/Fragment Test Series

by

T. G. George, R. E. Tate, and K. M. Axler

ABSTRACT

The radioisotope thermoelectric generator (RTG) that will provide power for space missions contains 18 General-Purpose Heat Source (GPHS) modules. Each module contains four $^{238}\text{PuO}_2$ -fueled clads and generates 250 W_(t). Because a launch-pad or post-launch explosion is always possible, we need to determine the ability of GPHS fueled clads within a module to survive fragment impact. The bullet/fragment test series, part of the Safety Verification Test Plan, was designed to provide information on clad response to impact by a compact, high-energy, aluminum-alloy fragment and to establish a threshold value of fragment energy required to breach the iridium cladding. Test results show that a velocity of 555 m/s (1820 ft/s) with an 18-g bullet is at or near the threshold value of fragment velocity that will cause a clad breach. Results also show that an exothermic Ir/Al reaction occurs if aluminum and hot iridium are in contact, a contact that is possible and most damaging to the clad within a narrow velocity range. The observed reactions between the iridium and the aluminum were studied in the laboratory and are reported in the Appendix.

I. INTRODUCTION

The General-Purpose Heat Source (GPHS) is a modular component of the radioisotope thermoelectric generator (RTG) that will provide power for a number of space missions. The first two flights will be the NASA Galileo and the ESA Ulysses (formerly International Solar-Polar) missions. The RTG generates electrical power by using the heat of ^{238}Pu α -decay to create a temperature differential across a thermoelectric array. The Galileo mission will require two RTGs and Ulysses will use a single RTG. Each RTG contains 18 GPHS modules.

Each fully loaded GPHS module contains four $^{238}\text{PuO}_2$ -fueled clads and provides a total thermal output of 250 W_(t) at the beginning of life. A clad consists of a fuel pellet encapsulated in a containment shell of an iridium-based alloy containing 0.3% tungsten (DOP-26). Two fueled capsules are held in a Fineweave-

Pierced Fabric* (FWPF) graphite impact shell (GIS), and two GISs are contained within the module's FWPF aeroshell (Fig. 1). Because a launch accident is always possible, the GPHS module has been designed to maximize the containment of plutonia during any accident.

In the Galileo and Ulysses missions, a fueled Centaur rocket will be transported within the cargo bay of the Space Transportation System vehicle (space shuttle). A launch explosion fueled by the shuttle and Centaur propellants could expose the RTG and GPHS modules to high overpressure and to a field of high-energy fragments. The bullet/fragment test series was included in the Safety Verification Test Plan¹ to provide information on GPHS module response to the impact of high-energy fragments and to establish a threshold value of

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

Fig. 1. Each fully loaded GPHS module contains four $^{238}\text{PuO}_2$ -fueled capsules.

fragment energy required to breach the iridium-alloy containment shell.

II. THE TEST PROGRAM

The SVT bullet/fragment tests series simulated the impact of compact fragments that a PuO_2 -containing GPHS module could experience in a launch explosion. We did not test GPHS modules containing fueled clads because we lack a test facility that could fully contain the highly toxic plutonium oxide. Instead, sintered uranium dioxide prepared from depleted uranium was used as the fuel simulant. The density and dimensions of the UO_2 pellets were tailored to match the mass and configuration of a plutonia fuel pellet. Although plutonium pellets are usually broken into several pieces by the time they have been loaded into the GPHS modules, the degree of fragmentation is quite variable, and we chose not to try to simulate it in these tests.

Before the simulant-fueled GPHS modules were tested, a short series of bullet/fragment tests was conducted to determine the attenuating effects of FWPF graphite. In these tests, 18-g .50-caliber aluminum-alloy bullets were fired into FWPF graphite plates at velocities of 320, 633, and 900 m/s (1050, 2076, and

2952 ft/s). The thickness of each FWPF graphite plate (4.86 mm) corresponded to the thinnest section of the GPHS module sidewall. The three tests are summarized in Table I. The test results indicate that while the stopping power of FWPF graphite is inversely related to bullet velocity, bullet deformation increases directly with increasing velocity. Note, however, that to impact a fueled clad within a GPHS module, a bullet/fragment would first have to penetrate two layers of FWPF graphite (aeroshell and GIS walls) and a layer of carbon-bonded carbon fiber (CBCF) insulation. While the attenuating effects of CBCF insulation are unknown (at 0.25 g/cm^2 and a thickness of 1.8 mm), multiple layers of FWPF graphite clearly would have a significant nonlinear effect on bullet velocity. The test results show that the bullet nose mushrooms after striking a single layer of FWPF; if a deformed bullet were to strike a second FWPF layer, the increased frontal contact area would proportionally increase the velocity decrement required to penetrate the second layer.

A. Components

The target assembly consisted of four parts: a FWPF half-module containing a FWPF GIS and two simulant

TABLE I. Attenuation of Bullet Velocity in FWPF Graphite^a

Bullet Velocity (m/s)		Velocity Decrement		Bullet Deformation		
Initial	Exit	(m/s)	(%)	Orig. Diam.	Max. Final Diam.	% Change
320	288	32	10.0	0.500	0.518	3.6
633	580	53	8.4	0.500	0.590	18.0
900	832	68	7.5	0.500	0.638	27.6

^aAs determined from the trajectories of 18-g .50-caliber aluminum-alloy bullets fired through a 4.86-mm FWPF graphite plate.

clads, a bulk graphite backing block, and two bulk graphite blocks of GPHS configuration adjacent to the target module (Fig. 2). The backing block and adjoining blocks were weighted with tantalum rods so that the total masses matched the masses of the components being simulated. The FWPF components were made from flight-quality material and were machined to flight configurations. The iridium-alloy containment shells were also flight-quality units provided by Mound Facility. Los Alamos fabricated the UO₂ fuel simulant pellets, welded the containment capsules, and assembled the target components. Data describing the iridium alloy and UO₂ test components are presented in

Table II; the simulant clads used in each test are listed in Table III.

B. Procedures

To simulate the impact of a high-energy, compact fragment on a GPHS module, we fired a .50-caliber aluminum-alloy bullet, which weighed 18 g and had a nonballistic configuration (Fig. 3), into the GPHS half-module. The bullet was fabricated from certified 2219-T87 aluminum alloy, the same material used for the space shuttle fuel tankage.

Fig. 2. The aluminum-alloy bullet was fired into a simulation of a module stack.

TABLE II. Components Used in the SVT Bullet Impact Tests

Capsule	Iridium Clad		UO ₂ Pellet ^a	Fueled Capsule Weight (g)
	Vent Cup	Blind Cup		
IRG-111	LR281-2	LR276-6	62-98	206.15
IRG-112	LR282-2	LR281-3	62-99	206.92
IRG-113	LR283-1	LR282-1	62-88	206.97
IRG-114	LR283-3	LR282-3	62-89	207.99
IRG-119	LR286-2	LR287-3	62-104	206.93
IRG-120	LR288-2	LR287-8	62-107	207.32
IRG-130	MER26-6	MER25-1	62-119	201.04
IRG-131	MER24-4	MER25-3	62-121	202.62

^aDepleted ²³⁸U.

The bullet/fragment tests were conducted at a Los Alamos outdoor test range. The gun that fired the projectiles was a thick-walled, .50-caliber barrel mounted on a heavy pedestal. The bullet velocity was modified as needed by adjusting the powder loading in the .50-caliber cartridge case and filling the remaining case volume with bran. The gun was aimed with a telescopic bore sight, the aim line was aligned with vernier handwheels, and the gun was triggered electrically by remote control from a firing bunker. For the GPHS bullet/fragment tests, the gun was aimed at the center of the target (Fig. 1).

For each test, the target assembly was heated in a wire-wound furnace insulated with asbestos and carbon felt. A similarly insulated quartz tube was mated coaxially to the furnace end with a graphite adapter ring. The test components were arranged so that the aim point of the .50-caliber gun coincided with the furnace axis. The quartz tube was an x-ray transparent chamber into

which the target assembly was withdrawn very shortly before the projectile was fired. A flash x-ray was triggered to record the bullet position at or just before impact with the target.

The four-component test assembly was positioned in the furnace section with the nonclosure side of the half-module target normal to the projectile path. Two type-K thermocouples were inserted in grooves in the weight-simulant modules, adjacent to the target clad. The components of the target assembly were bound with a stainless steel clamp to maintain their relative positions when the assembly was moved into the quartz tube. Argon gas was piped into the furnace and quartz chamber at about 140 l/h to protect the test articles and the graphite components of the furnace during the heat-up and test periods and during the early portion of the cool-off period.

The target module was heated to 1125°C and held there for 15 min. Then, as quickly as possible to avoid module heat loss, the module was retracted into the quartz chamber, the furnace plug was removed to clear the projectile path, and all personnel withdrew to the firing bunker. The gun was fired as soon as practical.

On the first two tests, the projectile velocity was measured with an Ohler velocity screen. For the three other tests, the velocity screen was not used because, to improve impact accuracy, the gun had been moved much closer to the target. Therefore, the projectile velocity was calculated from a combination of flash x-ray timing and the location of the bullet on the x-ray film.

After the bullet had been fired, furnace power was disconnected, the furnace plug was replaced, and the argon gas flow was maintained for about 3 h. The furnace and target components were allowed to cool overnight, and on the following morning the target was

TABLE III. Clads Used in the SVT Bullet Impacts

Test No.	Target Clad ^a	Secondary Clad
1	IRG-113	IRG-114
2	IRG-119	IRG-120
3	IRG-130	IRG-131
4	IRG-111	IRG-112
5	IRG-112	IRG-131

^aIn each test, the target clad was loaded into the blind end of the graphite impact shell and was positioned at the closure end of the half-module.

Fig. 3. An 18-g 2219-T87 bullet was selected to represent a typical high-energy fragment; all dimensions are in inches.

removed from the quartz tube section and radiographed. The target was then transported to the metallographic laboratory for disassembly and postmortem examination.

III. RESULTS

The results and conditions of each bullet/fragment test are summarized in Table IV. The target module was disassembled and photographed after it was received at the metallographic laboratory. The simulant-fueled clads were removed from the half-module, examined, and photographed. The diameter and length of each clad were measured to determine the capsule deformations. Postimpact strains of the target clads are listed in Table V; the secondary clads experienced no deformation and were occasionally reused in later tests. After capsule measurement, specimens for metallographic examination were cut from the target clad vent and weld-shield cups. Grain sizes of the iridium cups are listed in Table VI; representative cup microstructures may be seen in Figs. 4-13. Details of the individual tests and postmortem examinations are given below.

A. Bullet/Fragment Test 1

In the first test, the velocity measured by the Ohler screens was 319 m/s (1048 ft/s), but a posttest examination of the furnace found a scar in the graphite that may have been caused by a glancing impact of the bullet before it hit the target. If that occurred, the velocity of the bullet when it hit the target would have been somewhat lower than the measured velocity. The temperature at impact was 1091°C. The bullet passed through the aeroshell, partially penetrated the GIS, and struck the target clad (IRG-113) approximately 3.5 mm above the weld centerline (weld-shield cup). A thin layer of powdered graphite was sandwiched between the bullet and clad, preventing contact of the aluminum and hot iridium. The bullet impact caused only minor clad deformation (Fig. 14); postimpact examination of the target clad did not reveal any cracks or areas of high local deformation. Microscopic examination of the capsule weld revealed an acceptable weld microstructure (Fig. 15).

TABLE IV. Summary of the SVT Bullet Impacts

Test No.	Temperature (°C)	Velocity (m/s)	Clad	Bullet Contact Area (mm ²)	Remarks
1	1091	319	IRG-113	93	Bullet struck the clad ≈ 3.5 mm above the weld centerline and did not fully penetrate the impact shell; no breach.
2	1030	415	IRG-119	288	Bullet struck the clad tangentially along the weld centerline; weld unzipped over 330°. Breaching crack measured 2.5 mm at widest point. Three small areas of Ir/Al reaction observed on clad surface.
3	1080	458	IRG-130	342	Bullet impacted ≈ 4.0 mm above the weld centerline, remained in place, and melted. Brittle intermetallic formed at Ir/Al interface. Large hole (10 mm $\times$ 3.5 mm) observed on vent-cup impact face.
4	1060	460	IRG-111	266	Bullet struck the clad ≈ 6.0 mm above the weld centerline; no breach. Two small areas of Ir/Al reaction observed on clad surface.
5	1120	555	IRG-112	169	Bullet impacted approximately 9.0 mm above the weld centerline; hairline crack on blind-cup radius (≈ 6.0 mm in length) and along weld centerline (≈ 4.0 mm in length). No evidence of Ir/Al reaction.

TABLE V. Postimpact Capsule Strains

Capsule No.	Bullet Test	Impact Velocity (m/s)	Vent Cup		Weld		Blind Cup		Max. Axial Strain (%)
			min	max	min	max	min	max	
IRG-113	1	319	0	0	-1.0	0	-3.4	0	0
IRG-119	2	415	-1.0	0	-3.0	+1.7	0	0	+10.7 ^a
IRG-130	3	458	-3.0	+2.0	-13.1	+6.0	-12.8	+4.4	+4.0
IRG-111	4	460	0	0	+2.0	+2.3	-11.1	+3.4	+4.0
IRG-112	5	555	0	0	-1.0	+2.3	-20.2	+2.3	+2.7

^aThe IRG-119 closure weld unzipped over 330° of the clad circumference.

**TABLE VI. Average Grain Sizes of the Iridium Cups
Used in the SVT Bullet/Fragment Tests**

Capsule	Iridium Cup	Average Grain Size ^a
IRG-113	Vent	24.5
	Weld Shield	24.3
IRG-119	Vent	23.8
	Weld Shield	23.5
IRG-130	Vent	21.7
	Weld Shield	22.8
IRG-111	Vent	23.3
	Weld Shield	22.8
IRG-112	Vent	23.0
	Weld Shield	19.0

^aBased on the average number of grains/0.635 mm nominal wall thickness.

Fig. 4. The IRG-113 vent cup had a fine-grained microstructure; etched, 50X.

Fig. 5. The microstructure of the IRG-113 weld-shield cup was similarly fine grained; etched, 50X.

Fig. 6. The IRG-119 vent cup also had a fine-grained microstructure; etched, 50X.

Fig. 7. The IRG-119 weld-shield cup had an average of 23.5 grains/0.635 mm nominal wall thickness; etched, 50X.

Fig. 8. The IRG-130 vent cup had a fine-grained microstructure; etched, 50X.

Fig. 9. The microstructure of the IRG-130 weld-shield cup was similarly fine grained; etched, 50X.

Fig. 10. The IRG-111 vent cup also had a fine-grained microstructure; etched, 50X.

Fig. 11. The IRG-111 weld-shield cup had an average of 22.8 grains/0.635 mm nominal wall thickness; etched, 50X.

Fig. 12. The IRG-12 vent cup also had a fine-grained microstructure; etched, 50X.

Fig. 13. The IRG-112 weld-shield cup was similarly fine grained; etched, 50X.

Fig. 14. In the first bullet/fragment test, the target clad (IRG-113) experienced only moderated deformation. Capsule IRG-113 is on the left (vent down), and IRG-114 (vent cup) is on the right; 1.5X.

Fig. 15. The IRG-113 closure weld had an acceptable microstructure; etched, 50X.

B. Bullet/Fragment Test 2

The bullet velocity was 415 m/s (1361 f/s), and the clad temperature was 1030°C. The temperature was lower because the first shot missed the target clad, allowing the target to cool before a second shot was fired. The bullet penetrated the GIS (Fig. 16) and struck the target clad (IRG-119) tangentially along the weld centerline. Although the target clad experienced only moderate deformation, the closure weld unzipped over a 330° arc (Fig. 17). The breaching crack measured 2.5 mm at the widest point. Macroscopic examination of the clad exterior revealed isolated areas of an apparent Ir/Al reaction (Fig. 18).

Specimens for metallographic examination were removed from the intact and fractured portions of the capsule weld. Examination of a section containing the crack terminus (Fig. 19) revealed a significant amount of porosity (Fig. 20) and an unacceptable weld microstructure (Fig. 21). The weld was centered approximately 0.5 mm above the cup interface and never achieved full penetration; apparently only 50% of the wall thickness had actually been welded. Examination of a section containing the widest portion of the breaching crack (Fig. 22), revealed a similar microstructure (Figs. 23 and 24).

Fig. 16. In the second test, the bullet penetrated the GIS and struck the target clad (IRG-119) at the weld centerline; 1X.

(a)

(b)

(c)

Fig. 17. The IRG-119 closure weld was unzipped over a 330° arc. (a) Impact face, (b) profile, and (c) vent end; all at 1.5X.

Fig. 18. Isolated Ir/Al reaction sites were visible on the IRG-119 vent cup; 1.5X.

Fig. 19. A transverse section of the IRG-119 closure weld was removed for metallographic examination; as polished, 7X.

Fig. 20. The IRG-119 closure weld contained a significant amount of porosity; as polished, 50X.

Fig. 21. The IRG-119 closure weld did not achieve full penetration; etched, 50X.

Fig. 22. A section for metallography was also removed from the widest portion of the IRG-119 weld failure; as polished, 7X.

Fig. 23. Insufficient penetration was also observed on the shield-cup side of the IRG-119 weld; etched, 50X.

Fig. 24. The IRG-119 weld failure had a brittle intergranular appearance; vent-cup side, etched, 50X.

C. Bullet/Fragment Test 3

For the third test, bullet velocity was increased to 458 m/s (1501 ft/s); clad temperature at impact was 1080°C. The bullet penetrated the aeroshell and GIS, and it struck the target clad (IRG-130) approximately 4.0 mm above the weld centerline (weld-shield cup). Capsule deformation was significant; the capsule was badly bulged at the closure weld (Fig. 25) and contained a centerline weld crack (approximately 22.0 mm in length) on the trailing face (Fig. 26). After impact, the bullet remained in contact with the clad and melted (Fig. 27). The molten aluminum reacted vigorously with the hot iridium, eroding a large hole (10.0 mm × 3.5 mm) into the vent-cup impact face (Fig. 28) and spattering reaction product across the clad exterior (Fig. 29). Although two holes were observed on the impact face, only the hole in the vent cup was an actual breach; the hole at the center of the bullet contact area was originally plugged by a deposit of Ir/Al reaction product. (The deposit was also fused to the solidified bullet and was broken away during module disassembly.)

Specimens for metallographic examination were removed from the capsule weld, from areas adjacent to the hole in the vent-cup impact face, and from Ir/Al reaction sites. Examination of a section containing the centerline weld crack revealed a coarse weld microstructure (Fig. 30). An intact section of the closure weld was

Fig. 25. Capsule IRG-130 experienced significant deformation; profile, 1.5X.

relatively fine grained (Fig. 31), but it had an unusual appearance.

Examination of a section adjacent to the hole in the vent-cup impact face revealed significant thinning of the capsule wall (Fig. 32). A similar effect was observed in sections adjacent to the hole at the center of the bullet contact area (Fig. 33). In this case, however, deposits of the Ir/Al reaction product were attached to the clad exterior (Figs. 34 and 35). The Ir/intermetallic interface was clearly defined, and the microstructure of the remaining iridium was apparently unaffected. The intermetallic compound appeared to be very homogeneous and was traversed by numerous fractures. A hardness survey across the Ir/intermetallic interface confirmed the lack of a transition zone and indicated that the intermetallic compound was nearly twice as hard as the parent iridium; details of the hardness scan are presented in Table VII.

A deposit of Ir/Al reaction product observed on the vent-cup radius (Fig. 36) was also sectioned for metallography. Microscopic examination of the cross-section revealed a narrow breaching crack (Fig. 37) and thick deposits of a homogeneous intermetallic compound (Figs. 38 and 39). As in previous samples, the intermetallic compound was badly fractured. The microstructure of the remaining iridium was apparently unaffected by the intermetallic reaction.

The deposit of Ir/Al reaction product that originally plugged the hole at the center of the bullet contact area was also examined metallographically and was submitted for microprobe analysis. Metallographic examination of a polished cross section (Fig. 40) revealed several distinct phases. Microprobe analysis of the same sample identified five phases containing iridium and aluminum (Table VIII).

The observed reaction between the iridium and the aluminum was obviously very rapid, so we studied it in the laboratory experiments reported in the Appendix.

D. Bullet/Fragment Test 4

The fourth test was intended to reproduce the conditions of the third test. The bullet velocity was 460 m/s (1508 ft/s), and the clad temperature at impact was 1060°C. The bullet penetrated the aeroshell and GIS, struck the target clad (IRG-111) approximately 6.0 mm above the weld centerline (weld-shield cup), and was deflected out of the aeroshell. Capsule deformation was moderate (Fig. 41). Macroscopic examination of the clad exterior did not reveal any cracks in the cup walls or closure weld. However, two small deposits of Ir/Al reaction product were observed on the surface of the weld-shield cup. The deposits apparently resulted from bullet segments that remained within the GIS and melted.

(a)

(b)

Fig. 26. A centerline weld crack was observed on the trailing face of capsule IRG-130; (a) 1.5X and (b) 5X.

Fig. 27. After impact, the bullet remained in contact with capsule IRG-130 and melted; 0.6X.

Fig. 28. The molten aluminum reacted vigorously with the hot iridium and eroded a large hole in the IRG-130 vent-cup impact face, 1.2X.

Fig. 29. Ir/Al reaction product was observed in numerous locations on the IRG-130 impact face; 1.5X.

Fig. 30. The centerline cracks in the IRG-130 closure weld occurred in a coarse-grained section; etched, 50X.

Fig. 31. The intact portion of the IRG-130 closure weld had an unusual microstructure; etched, 50X.

(a)

(b)

Fig. 32. Significant thinning was observed in a wall section adjacent to the hole in the IRG-130 vent cup. (a) As polished and (b) etched; 50X.

Fig. 33. Thinning was also observed in wall sections adjacent to the hole at the center of the bullet contact area; as polished, 7X.

(a)

(b)

Fig. 34. A thick deposit of Ir/Al reaction product was observed on the vent-cup side of the hole at the center of the bullet contact area. (a) As polished and (b) etched; both at 50X. (Note that hardness indentations are visible on the etched cross section).

(a)

(b)

Fig. 35. The shield-cup side of the hole was also coated with a thick deposit of Ir/Al reaction product. (a) As polished and (b) etched; both at 50X.

TABLE VII. Hardness Profile of the Iridium/Intermetallic Interface (IRG-130)

Location	Distance from Interface (in.)	DPHN (50-g load)
Intermetallic	+0.25	1121
Intermetallic	+0.20	1038
Intermetallic	+0.15	1084
Intermetallic	+0.05	946
Interface	0	721
Iridium	-0.05	418
Iridium	-0.10	412
Iridium	-0.15	521

Fig. 36. A deposit of Ir/Al reaction product on the IRG-130 vent-cup radius was also sectioned for metallography; 10.5X.

Fig. 37. The intermetallic deposit on the IRG-130 vent-cup radius ringed a narrow crack; as polished, 7X.

(a)

(b)

Fig. 38. The wall section on the radius side of the crack had been thinned significantly. (a) As polished and (b) etched; both at 50X.

(a)

(b)

Fig. 39. The microstructure of the remaining iridium was apparently unaffected by the Ir/Al reaction. (a) As polished and (b) etched; both at 50X.

Fig. 40. A deposit of Ir/Al intermetallic that originally plugged the hole at the center of the bullet contact area was sectioned for metallographic and microprobe analyses; as polished, 40X.

TABLE VIII. Phases Identified in an Intermetallic Deposit Removed from Capsule IRG-130^a

4% Ir: 96% Al
 15% Ir: 46% Al: 31% Cu: 8% Fe
 53% Ir: 47% Al
 62% Ir: 38% Al
 70% Ir: 30% Al

^aAll compositions in wt%. All phases identified by electron microprobe analysis.

(a)

(b)

(c)

Fig. 41. Capsule IRG-111 experienced only moderate deformation. (a) Impact face, (b) profile, and (c) trailing face; all at 1.2X.

The IRG-111 closure weld was sampled for comparison with previous capsule welds. Metallographic examination of the weld cross section (Fig. 42) revealed a slight wall misalignment and an unusual weld microstructure. The appearance of the weld microstructure indicated that full penetration had been barely achieved.

E. Bullet/Fragment Test 5

For the fifth test, bullet velocity was increased to 555 m/s (1820 ft/s); clad temperature at impact was 1120°C. The bullet penetrated the aeroshell and GIS, struck the target clad (IRG-112) approximately 9.0 mm above the weld centerline (weld-shield cup), and was deflected out of the aeroshell. Although the overall clad deformation was minor, localized deformation occurred on the radius of the weld-shield cup (Fig. 43). Macroscopic examination of the clad exterior revealed a small crack (approximately 0.2 mm $\times$ 4.5 mm) on the shield-cup radius, just beyond the edge of the bullet contact area. In addition, a small centerline weld crack (3.6 mm in length) was observed on the impact face of the clad.

The cracked portion of the capsule weld and the weld-shield cup radius were sectioned to provide specimens for metallographic examination. Examination of a transverse weld section revealed that the centerline crack penetrated more than 80% of the total wall thickness (Fig. 44). The weld microstructure was very coarse; at the centerline the weld contained only 3 grains/thickness.

Examination of the crack on the shield-cup radius revealed a jagged, intergranular fracture (Fig. 45). The breach occurred in a relatively fine grained section and apparently resulted from a stress overload; several smaller cracks were visible on the exterior and interior surfaces.

IV. DISCUSSION

The results of the postmortem examinations indicate a direct correlation between bullet velocity and clad damage. The postimpact capsule strains (Table V) increased with each increment of bullet velocity. Test temperatures varied, ranging between 1030°C and 1120°C. Test responses were not significantly affected, however, because this range was sufficiently close to the 1091°C intended temperature.

The degree of capsule damage was often affected by factors other than bullet velocity, such as impact angle, impact location, and Ir/Al intermetallic reactions. It was apparent, however, that real clad damage (deformation and fracture caused only by the bullet impact) increased with increasing bullet velocity.

The breach of capsule IRG-119 (bullet/fragment test 2) demonstrated the vulnerability of a capsule weld to impacts at the weld centerline (the bullet in test 2 struck the clad tangentially along the weld centerline). Although examination of the IRG-119 weld revealed incomplete penetration (Fig. 20) and a poor weld microstructure (Fig. 21), it is possible that even a good quality weld might have not survived the impact.

Fig. 42. The IRG-111 closure weld had an unusual microstructure; etched; 50X.

(a)

(b)

Fig. 43. Capsule IRG-112 experienced severe localized deformation on the radius of the weld-shield cup. (a) Impact face and (b) profile; both at 1.5X.

(a)

(b)

Fig. 44. The IRG-112 closure weld had a very coarse microstructure and contained a centerline crack that penetrated more than 80% of the wall thickness. (a) As polished and (b) etched; both at 50X.

(a)

(b)

Fig. 45. The fracture on the IRG-112 shield-cup radius had a brittle intergranular appearance. (a) As polished and (b) etched; both at 50X.

Metallographic examination of the other capsule welds revealed that three were of questionable quality (IRG-130, IRG-111, and IRG-112). All of the weld microstructures were very coarse, and the IRG-111 weld also had a problem with full penetration (Fig. 42). In addition, the IRG-111 weld bead bridged a significant wall misalignment. The welds of all five test clads were not of flight quality. With the exception of capsule IRG-119, all of the weld fractures could be related to microstructure or technique.

All of the iridium cups had fine-grained microstructures (Figs. 4 through 13). The average grain sizes ranged from 19.2 to 24.5 grains/0.635 mm nominal wall thickness. The performance of the iridium cups used in the bullet-fragment test series should be considered representative of the flight-quality clad response.

The small deposits of Ir/Al reaction products observed on capsules IRG-119 and IRG-111 (tests 2 and 4) and the condition of capsule IRG-130 (test 3) indicate that a vigorous Ir/Al reaction is possible in an impact of an aluminum projectile. The Ir/Al reaction appears to be most damaging when the impacting fragment has sufficient velocity to penetrate the graphite components, but not enough to allow it to continue out of the module. Because the Ir/Al reaction appears to require molten aluminum, any environment that significantly lowers the clad surface temperature (such as submersion in water) would prevent the reaction. Although the reaction (once initiated) is exothermic and disastrous to the iridium clad, it may be relatively benign in terms of fuel release because the Ir/Al reaction product adheres to the reaction site and tends to cover all of the surface details (such as cracks). However, the reaction product is very brittle, and it would be easily fractured by any additional deformation. An in-depth discussion of the intermetallic properties, possible Ir/Al phases, and Ir/Al thermodynamics is presented in the Appendix.

The velocity of the bullet in the fifth test (555 m/s) is apparently at or near a threshold value that will consistently produce severe localized deformation and subsequent breaching cracks. Although impact against a cup face undoubtedly requires more velocity to produce a breaching crack (the bullet in test 5 impacted at the weld-shield-cup radius), a radius impact is not improbable and is thought to be less severe than an end-on impact. Therefore, 555 m/s may be the fragment velocity that will produce a breaching crack in most impacts, but the question of fragment velocity required to break the clad in an end-on impact can only be answered with further tests.

V. CONCLUSIONS

- (1) The threshold for direct mechanical failure of a GPHS fueled clad impacted by an 18-g aluminum bullet is approximately 555 m/s.
- (2) When an aluminum fragment remains in contact with the clad for more than a few milliseconds, a chemical reaction occurs between aluminum and iridium, which is rapid enough to compromise the GPHS postfragment containment capability.
- (3) The weld quality in four of the five capsules tested was questionable. We believe that the weld failures seen in the tests at 458 and 555 m/s would not have occurred in flight-quality welds.
- (4) We are uncertain of the probable response in the 414 m/s test if the weld had been flight quality. The particular deformation in this test resulted in tensile stress across the weld that might have been sufficient to cause failure in a good weld.
- (5) All of the iridium clads had microstructures typical of those in flight-quality heat sources that may be exposed to accidents.

VI. RECOMMENDATIONS

- (1) The chemical effects of aluminum fragments on the iridium clad can be severe, as shown in these tests. The effects of fragments of other elements that are present on the space shuttle should be determined. Titanium, in particular, would be a good candidate for study, because it is both a common aerospace material and it might be expected to react with iridium.
- (2) The target orientation was fixed in these tests. However, an orientation that places the end of the capsule toward the direction of fragment travel is equally likely. This orientation should be investigated in future fragment tests.

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REFERENCE

1. "GPHS Safety Verification Test Series Procedure Manual," Los Alamos National Laboratory document MST-5-C-83-19 (April 14, 1983), Section 5.2.2. (launch accident, fragment impact).

APPENDIX

STUDIES OF THE Ir/Al SYSTEM (K. M. Axler)

I. INTRODUCTION

In earlier studies, researchers^{A-1} produced arc-melted samples with compositions ranging from 1:2 to 1:4 atomic ratio Ir/Al. Powder photographs of these Ir/Al samples revealed strong similarities to the corresponding compositions in the Os/Al system. These correlations^{A-2} refer to Os₂Al₃, OsAl₂, OsAl₃, and Os₄Al₁₃. Researchers^{A-3} in Italy have reported the existence of the phases IrAl, IrAl_{2.7} (cubic), IrAl₃ (hexagonal), IrAl_{3.75}, and IrAl₅.

The present work described here is a project directed at characterizing the reactions within the Ir/Al system and determining the properties of the system with respect to the radioisotopic heat source program. This work has involved the development of laboratory techniques to prepare samples within the Ir/Al binary system and the analysis of these samples for their composition and structure. Currently, we have produced a series of samples ranging from 1:7 to 3:1 atomic ratio Ir/Al. Analyses by x-ray diffraction and electron microprobe were used to determine the phases produced. Melting points and heats of reaction are being determined by differential thermal analysis.

In our current studies, we have confirmed the existence of a series of stable compounds in the Ir/Al binary system. Contact of aluminum with iridium at temperatures as low as 660°C (the melting point of aluminum) produces an exothermic reaction. The subsequent heat generated accelerates the reaction and the consumption of iridium results in a region of intermetallic phases.

II. SAMPLE PREPARATION

The samples produced to date comprise the Ir/Al atomic ratios 3:1, 1:1, 2:3, 1:2, 1:2.6, 1:3, 1:4, 1:5, and 1:7. We have used two different techniques for sample preparation. One technique is arc melting in an inert

atmosphere. In the arc-melting procedure, the materials we used were iridium tube and aluminum sheet. The aluminum was commercially available with a purity of 99.999%. The iridium was of high purity; an in-house analysis reported all impurities to be below their respective detection limits.

In the arc-melting procedure, materials were melted in the inert atmosphere before they were reacted. Previous work^{A-4} has shown a similar technique to be effective in removing oxygen from both iridium and aluminum. This is done to provide the most pure samples possible and to avoid any possible inhibiting effect of an oxide coating on reactivity.

Another technique used in the preparation of other samples was vacuum heating in a radio-frequency induction heater. This technique enabled us to simultaneously heat samples of varying composition, thus assuring identical reaction conditions. For the induction heating, the materials used are high-purity iridium and aluminum powders. All annealings are done in evacuated quartz glass tubes lined with tungsten foil to prevent any possible interactions between the samples and the quartz.

III. EXPERIMENTAL RESULTS

Arc melting of the 1:1 atomic ratio produced the intermetallic compound, IrAl. Analysis by x-ray diffraction confirmed the presence of cubic IrAl, Pm3m, type crystal structure, $a = 2.98 \text{ \AA}$, with Ir at 0 0 0 and Al at $\frac{1}{2} \frac{1}{2} \frac{1}{2}$. We have also formed the IrAl intermetallic compound in the differential scanning calorimeter (DSC). The DSC revealed an onset of reaction at the melting point of aluminum. Using calorimetry, we obtained a value of -36.9 kcal/mol for the heat of formation of IrAl. A theoretical value of -33.6 kcal/mol was calculated using the technique developed by Miedema.^{A-5} Another

experiment we performed involved the heating of an IrAl sample under inert gas to determine a melting point. The sample was brought up to 1500°C without melting. This sample's resistance to melting is consistent with the thermodynamic stability we had determined.

Arc melting of the 1:3 composition produced a combination of two phases, IrAl and IrAl₃. The IrAl₃ phase was a cubic A15 type structure with $a = 7.60 \text{ \AA}$. Iridium is at the 0 0 0 and $\frac{1}{2} \frac{1}{2} \frac{1}{2}$ positions. The aluminum atoms are at the $\frac{1}{4} 0 \frac{1}{2}$, $\frac{1}{2} \frac{1}{4} 0$, $0 \frac{1}{2} 14$, $\frac{3}{4} 0 \frac{1}{2}$, $\frac{1}{2} \frac{3}{4} 0$, and $0 \frac{1}{2} \frac{3}{4}$ positions. This phase may be a high-temperature polymorph of the hexagonal IrAl₃ reported by Ferro and coworkers.^{A-3} The arc-melted sample was in a 10-20% ordered crystalline condition. A section of this sample is being annealed at 800°C and will again be analyzed by x-ray diffraction. Interestingly, this cubic structure is analogous to IrMo₃,^{A-6} IrNb₃,^{A-7} IrTi₃,^{A-8} and IrV₃,^{A-9, A-10, A-11} and it describes a diagonal trend in the periodic table.

The 1:2.6 atomic ratio Ir/Al was reacted by arc melting. Upon analysis, the product phase produced a unique x-ray pattern consisting of lines not associated with IrAl or IrAl₃. This is possibly the IrAl_{2.7} phase reported by Ferro and coworkers.^{A-3} The material is being annealed before a re-examination by x-ray diffraction. A section of the sample will also be submitted for electron microprobe analysis to quantitatively determine its components.

Another arc-melted sample was reacted consisting of a 3:1 atomic ratio Ir/Al. This reaction produced a mixture of the IrAl intermetallic phase with excess iridium, suggesting the absence of any compounds on the iridium-rich side of the system. The possibility still remains that a low-temperature, stable, iridium-rich compound may exist, which is incongruently melting and did not appear due to rapid cooling. To verify the absence of an iridium-rich compound, the sample is being annealed at 800°C and will again be submitted for x-ray diffraction analysis.

An experiment is underway to react a powder mixture of 1:5 Ir/Al in the DSC in an effort to produce the IrAl₅ phase and determine its heat of formation. A sample richer in aluminum (1:7 atomic ratio) is being prepared by arc melting. We will anneal this sample and then have it analyzed by x-ray diffraction. By providing an excess of aluminum and achieving equilibrium through annealing, we will confirm the intermetallic compound highest in aluminum content (currently thought to be IrAl₅).

IV. FUTURE Ir/Al STUDIES

Utilizing our available equipment and resources, we will define and characterize the complete set of compounds within the Ir/Al binary system with respect to their potential role in heat source applications. With the projected information obtained, a complete binary phase diagram will be determined for the Ir/Al system.

Previous research^{A-12} has shown that specimens of 1:1, 1:2, and 1:4 atomic ratio Ir/Al exhibit oxidation-resistant characteristics. The potential for Ir/Al intermetallics as oxidation-resistant coatings deserves attention. The high-temperature capabilities of our differential scanning calorimeter and thermal gravimetric analyzer are ideally suited for this study. We will produce the Ir/Al intermetallic compounds and then analyze them for reactions with oxygen at temperatures up to 1500°C. The possible use of an intermetallic compound as a prime cladding material also deserves consideration. Our current studies show good thermodynamic stability for the Ir/Al intermetallics. The mechanical properties of the compounds will be examined for such possible applications.

The heat produced by the formation of Ir/Al compounds would considerably raise the temperature of the heat source cladding. This in turn would further drive the Ir/Al reactions and possibly expose the fuel. The compatibility of the Ir/Al phases with the heat source fuel should be studied; it is presently known that plutonium forms ternary oxides containing plutonium and aluminum (for example, PuAlO₃).^{A-13} This study would involve the formation of any ternary oxides with plutonium and aluminum and possible vapor transport reactions with the fuel.

REFERENCES

- A-1 A. Magneli, "Quarterly Report on Contract DA-91-591-EUC-3134," Defense Technical Information Center report AD 449697 (1964), p. 4.
- A-2 A. Magneli, L. E. Edshammar, T. Dagerhamn, and S. Westman, "Final Technical Report on Contract DA-91-591-EUC-3134," Defense Technical Information Center report AD 451442 (1964), pp. 17-25.
- A-3 R. Ferro, R. Capelli, R. Marazza, S. Delfino, *Rend. Sc. Fis. Mat. E. Nat.* 45, 556-563 (1963).

- A-4 L. Brewer and G. Rosenblatt, *Trans. AIME* 224, 1260-1271 (1962).
- A-5 A. R. Miedema, "Plutonium 1975 and Other Actinides," in *5th International Conference on Plutonium and Other Actinides*, H. Blank and R. Lindner, Eds. (North-Holland Publishing Company, Amsterdam, 1976), pp. 3-11.
- A-6 E. Raub, *A. Metallkunde* 45, 23-30 (1954).
- A-7 S. Geller, B. T. Matthias, and R. Goldstein, *J. Am. Chem. Soc.* 77, 1502-1504 (1955).
- A-8 S. Geller, *Acta Cryst.* 9, 885 (1956).
- A-9 B. T. Matthias, S. Geller, and E. Corenzwit unpublished work quoted in S. Geller, *Acta Cryst.* 9, 885 (1956).
- A-10 M. V. Nevitt, *Trans. AIME* 212, 350-335 (1958).
- A-11 A. G. Knapton, *J. Inst. Metals* 87, 28-32 (1959).
- A-12 H. E. N. Stone, *J. Mater. Sci.* 10, 923-934 (1975).
- A-13 L. E. Russell, J. D. L. Harrison, and N. H. Brett *J. Nucl. Mater.* 2,4; 310-320 (1960).