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Compiled by

S. E. Bronisz
ABSTRACT

This formal monthly report covers the studies related to the use of $^{238}$PuO$_2$ in radioisotopic power systems carried out for the Space and Terrestrial Systems Division of the U. S. Department of Energy by the Los Alamos National Laboratory.

Most of the studies discussed here are of a continuing nature. Results and conclusions described may change as the work continues. Published reference to the results cited in this report should not be made without the explicit permission of the person in charge of the work.
I. GENERAL-PURPOSE HEAT SOURCE

A. Impact Tests (R. Zocher)

As we have indicated previously, the results of the non-closure, side-on impact tests IRG-88 and -90 make it imperative that an impact test with the heat source module in the large face-on orientation be performed. In January, Los Alamos issued a test plan for a fifth Design Verification Test to extend the previous verification series to the face-on orientation. Since then, the power supply systems contractor, General Electric Company, has introduced two design changes whose purpose is to increase the impact temperature of the module to 892 from 850°C. These changes consist of the application of a 0.51-mm-thick pyrolytic graphite coating to the corners of the impact shell and the provision for a 0.05-mm axial gap between the impact shell and the re-entry shell.

The design changes, with the accompanying impact temperature increases, are significant enough to require a new verification test series, which is referred to as the Design Iteration Test series. Accordingly, a test plan was written and issued (CMB-5-C-81-22, February 26, 1981) for the first of these impact tests, DIT-1, which will be in the face-on orientation. The schedule from that test plan is shown in Fig. 1. Its timely completion relies heavily on the receipt of the modified impact shells and the fueled clads.

B. Iridium Grain Growth Experiments

In the course of a study to determine the effects of small quantities of phosphorus on the grain growth of DOP-26 iridium, it was observed that a P-doped sample annealed at 1330°C for 500 h had a grain size consistent with previously obtained results, while an identically doped sample annealed for 2000 h had a finer grain size than predicted from the previous work. Samples of each, along with an as-received sample were sent to Oak Ridge National Laboratory for Spark-Source Mass Spectroscopy analysis to compare the thorium contents and to resolve whether or not all samples were indeed DOP-26 iridium. The results of the analyses indicated the thorium contents of all specimens were within the specification limits for DOP-26. Those elements that varied among the samples are listed in Table I.

The principal difference between the 500-h sample and the 2000-h sample appears to be the tungsten content. Further controlled studies would be required to determine whether or not the tungsten content is an important factor in the grain growth kinetics of P-doped DOP-26 iridium.
C. Iridium Fabrication (F. W. Schonfeld)

An additional iridium cup was formed from blank M-429 at 1000°C in the Los Alamos vacuum drawing equipment, which features a molybdenum alloy (TZM) die set with a Si₃N₄ draw ring and punch insert. The drawing conditions were:

- **Atmosphere**: vacuum, 10 μm
- **Temperature**: 1000°C
- **Pressure, holddown**: 4 MPa
- **Pressure, drawing**: 12 MPa, peak
- **Velocity**: 1.3 mm/s

The Si₃N₄ drawing broke during the draw, so that the upper side wall was not ironed and contained much more wrinkling than the cups drawn previously. The radius region was within specification and defect-free. This can be seen in Fig. 2. The microstructure of the drawn cup is shown in Fig. 3. The strains measured from the circle grid were quite similar to those measured on cups made by Mound Facility and Oak Ridge National Laboratory, indicating that none of the drawing processes places less severe demands on the iridium.

D. Laser Marking of Iridium (F. W. Schonfeld)

To provide continuous traceability of components, Mound Facility has proposed that incoming iridium blanks be marked with serial numbers applied by a laser ablation method. In support of this effort, Los Alamos treated two pairs of iridium disks in biaxial tension at 965°C and 45 mps. One pair was in the fibrous, wrought condition and the other pair was in the recrystallized condition after a 19-h, 1500°C heat treatment. One of each pair was marked with an array of laser-etched numbers and all disks were circle gridded for strain measurements. The unmarked disk was used as a control sample for each pair.

1. Fibrous structure. The two unrecrystallized iridium disks were designated L252-1, unmarked control, and LR280-2, laser numbered. Their maximum strains were comparable, ~26%. Figure 4 shows the two tested disks; some of the numbers etched with the laser are visible in Fig. 4b. Because of the fibrous structure, the failures were accompanied by extensive delamination, as shown in Fig. 5.

   The numbers on the marked sample were etched to varying depths. An example is the "2," which may be seen in the 3 o'clock position in Fig. 6. Its presence perturbed the crack path, as can be seen in Fig. 6a. At high magnification, Fig. 6b, the limited depth of perturbation can be seen.

2. Recrystallized structure. The two recrystallized iridium disks were designated L281-8, unmarked control, and L249-5, laser numbered. As in the disks with the fibrous microstructures, the maximum strains in this pair were similar, 14% for the control disk and 15% for the laser marked disk. The as-tested disks are shown in Fig. 7.

   The microstructure of the recrystallized control disk is shown in Fig. 8. The fracture mode was primarily intergranular, as can be seen in Fig. 9.

   The laser-marked disk had the same grain structure and also fractured intergranularly, as shown in Fig. 10. The fracture definitely appeared to be affected by the laser-etched numbers. Figure 11 shows the failure in the region of the top of the "2" visible in the approximate polar location in Fig. 7b.

   Figure 12 shows the second "2," which is visible in the 9 o'clock position of the dome in Fig. 7b. Two cracks were associated with this "2," one along its base, which was on a latitude line, and the other with its stem. At higher magnification
the nature of the cracks is clearly visible (Fig. 13), as is the irregular nature of the depth of the laser-induced pits.

It is evident that the laser marking procedure that was used on these disks resulted in some marks that were deep enough to be significant stress raisers. The numbers etched into the test disks were not all the same depth and, as has been seen, the depth can vary from one laser pulse to the next. It is evident that more work must be done before laser-marking of iridium can be approved for use. It would be necessary to determine how deep an etch is permissible, i.e., what depth does not affect the failure strain and is still visible after processing. Two marking parameters affect legibility, the actual depth of the individual pits and their spacing. It is possible that disconnected pits would be superior to connected ones from the standpoint of crack nucleation. It is evident that part of any such study should include an evaluation of other marking techniques, such as hand scribing.

II. SYSTEMS SUPPORT

A. Stirling Isotope Power System (D. Pavone)

The results of the visual examination of the components from the 800°C test assembly were reported last month.

Metallographic examinations of samples of the plutonia sphere revealed the existence of a duplex microstructure with a high-density crust and a low-density core, as illustrated in Fig. 14. The microstructure of the crust was characterized by small, uniformly distributed pores and the presence of grain boundary helium bubble strings. The core region, however, consisted of large loosely sintered, high-density, angular shards and smaller particles, as shown in Fig. 15. In contrast to the crust region, no grain boundary helium bubbles were observed in the core sample. Only very minor quantities of a dark etching phase that could be interpreted as a (Pu,0,C) ternary compound were detected.

Metallographic examination of cross sections through the vent assemblies showed the deposition of a nonmetallic material at the entrance of the vent-hole. This material, illustrated in Fig. 16, was identified as MgO by electron microprobe analysis.

A summary of results of other analyses follows.
1) The helium inventory of the plutonia sphere was 0.139 cc/g.
2) The plutonium content of the shell was 1.2 μg.
3) The phosphorous content of the plutonia was 5 ppm.
4) The phosphorous content of the shell was equivalent to 42 ppm.
5) Major impurities in the plutonia were Mg-300 ppm, Ca-500 ppm, Fe-170 ppm, Si-110 ppm, and Ta-400 ppm.
6) Principle components of the light gray deposit found at the entrance to the Inconel tube were magnesium, aluminum, silicon, chromium, cobalt, nickel, and molybdenum.
7) The grain sizes of the iridium hemishells were 19.0 and 22.1 grains/thickness.

B. Multihundred Watt

Fuel sphere assembly MHFT-74 was aged 30 days at 1210°C in vacuum and impacted under orbital-decay re-entry conditions, i.e., a re-entry pulse to an iridium temperature of 1500°C and an impact temperature of 1430°C. The impact velocity was 81 m/s.

The impact occurred with the graphite impact shell oriented at 180°C to the initial point of impact. The shell was split and delaminated.

Post-aging radiography revealed that the plutonium sphere was badly fractured, as shown in Fig. 17. The impact of the fuel clad occurred with the weld bead of the
iridium shell oriented nearby parallel to the granite target. No fractures or fingerprint cracks of the iridium were observed. Photographs of the impacted fueled clad are shown in Fig. 18. While fuel fragment punch displacements were visible on the impact face, severe local deformation of the iridium did not occur.

The post-impact diameter was 43.54 mm and the height was 29.74 mm. Based upon a nominal diameter of 40.64 mm, the diametral strain was +7.1% and the height strain was -26.8%.

Metallographic examination of a cross section including the weld and portions of each hemisphere showed that the microstructure of the weld bead was excellent, as shown in Fig. 19. Though visually the weld band appeared to be intact and no gross iridium transport was observed, microscopic examination of the interior surface of the post-impact containment shell showed the presence of a thin layer of iridium partially detached from the shell, as shown in Fig. 20. The morphology of this iridium layer differs from the columnar structure usually associated with the iridium transport phenomena; however, the tendency to be only partially bound to the iridium shell is also at variance with prior observations of flat surface grains on the interior surface of iridium exposed to plutonia. Evidence that suggests an iridium transport mechanism is shown in Fig. 21 in which iridium particles are seen intimately mixed with vapor deposited plutonia on the surface of the inner baffle of the vent directly behind the weld band.

The grain size of the iridium hemishells, as determined by the grain intercept technique, was 8.5 and 9.1 grains/thickness. This grain size is larger than observed for other FSAs with this aging treatment as well as being larger than the grain size predicted by our P-doping experiments.

Analysis of the recovered impact shell debris indicated the presence of 25.1 µg of plutonia and a phosphorous content equivalent to 73 ppm, based upon a nominal 120 g of iridium shell.

The phosphorous content of the plutonia sphere was found to be 5 ppm. This result is consistent with the pretest γ-scan result of 20 ppm. Photomacrophotographs of samples of the plutonia sphere from the impact face and the core exhibiting extensive fracturing are shown in Fig. 22. A region of extremely fine particles in the core sample is shown at higher magnification in Fig. 23. The presence of second-phase impurity particles is shown in the photographs of Fig. 24. None of these impurity particles was observed in the core sample.

Emission spectrographic analysis of a sample of the plutonia sphere indicated the major impurities to be: Ca-0.1%, Si-460 ppm, Mg-400 ppm, Fe-160 ppm, and Al-35 ppm.

The chemical analysis and photomicrographic evidence suggest a nonhomogeneous distribution of impurities, and illustrate the difficulty in assessing the purity of the plutonia by analyses of small samples.

III. LIGHT-WEIGHT RADIOISOTOPIC HEATER UNIT

A. Production (R. A. Kent)

Calorimetric measurements were completed for 34 of the Light-Weight Radioisotope Heater Unit pellets encapsulated in December. These data, together with other nondestructive test data, are listed in Tables II-IV. When all the calorimetric measurements have been made, the data will be corrected for decay and normalized to a common date.

B. Graphite Components (R. E. Tate)

The status of the manufacture of the tubular pyrolytic graphite insulator bodies was reviewed with D. Gabriel and N. Hignite of Mound Facility. One-third of
the parts machined fall out of specification for lack of minimum standoff rings. Lack of concentricity and delamination of the stock are the major sources of the defect. Further relaxation of the standoff requirements is not acceptable for re-entry thermal reasons. Some suggestions were made for the procurement of better stock material and improvements in the machining practice to achieve higher yields in producing the remainder of the required pieces.

C. Acceleration Forces (R. E. Tate)

More prints detailing the Light-Weight Radioisotope Heater Unit holding bracket for the Galileo Probe were received from General Electric. This holding bracket will be used for high-G load testing of the light-weight radioisotope unit. However, suitable self-locking nuts, torque specifications, a mutually agreeable method of interfacing the convex surface of the bracket with a centrifuge adapter plate, and detailed test parameters are still lacking. J. Terhune of NASA-Ames will pursue resolution of these details.

D. Cement Outgassing (R. E. Tate)

A 2.5-g batch of the aeroshell cement, UCAR C-34, was prepared, applied to aluminum foil as 0.1-g samples, and cured at 130°C as specified by Union Carbide. These samples are now ready to be installed in a vacuum manifold for weight-loss determination.

E. Postmortems (C. C. Land)

Five example and certification welds of light-weight radioisotope unit capsules were examined metallographically. All welds were good and showed complete penetration. In two cases, the weld shim was partially incorporated in the closure weld.

IV. SAFETY TECHNOLOGY

A. Helium Release (C. C. Land)

A $^{238}$PuO$_2$ pellet that was stored 7 y at ambient temperature was sectioned longitudinally and examined metallographically after it had been given serial temperature pulses of 1000, 1200, 1400, and 1600°C. There were no fine helium bubbles in the grain boundaries, but there was coarse interparticle porosity, which probably resulted from diffusion and coalescence of helium bubbles in those areas.

B. Enhanced Ductility Fuel (C. C. Land)

Eight cold-pressed and sintered pellets in the enhanced-ductility series were examined microscopically after they had been sectioned, polished, and etched.

Three $^{239}$PuO$_2$-1.3 mol% Nb$_2$O$_5$ pellets were found to contain 2-5% of Nb$_2$O$_5$ as a second phase, suggesting that it has a very limited solubility in the fluorite structure of plutonium oxide.

One of the $^{239}$PuO$_2$-1.3 mol% La$_2$O$_3$ pellets examined was found to be single-phased and the other contained ~1% of a second phase.

Two of the three $^{239}$PuO$_2$-1.3 mol% Y$_2$O$_3$ pellets were found to be single phased, but the third contained 3% of a second phase.

C. Test Pellet Fabrication (R. A. Kent)

Nine 15-W pellets were fabricated for environmental tests. All fabrication parameters were the same as those used for both the Light-Weight Radioisotope Heater Unit and the General-Purpose Heat Source pellets. The pellets were hot pressed from
Los Alamos GROG-type feed granules (83.5 at.% $^{238}$Pu) seasoned at 1100°C (60 wt%) and 1600°C (40 wt%). After hot pressing, the pellets were sintered for 6 h at 1527°C in Ar-H$_2^{16}$O. The data for these pellets are summarized in Tables V through VII.

V. FUEL PROCESSING

Two lots of Savannah River Plant feed powder (0.4 kg) were processed at Los Alamos. The material was $^{16}$O-exchanged, ball-milled and then slugged and screened to form <125-μm granules. The granules were then seasoned in Ar-H$_2^{16}$O at 1100°C (60 wt%) and 1600°C (940 wt%) and used to fabricate the environmental test pellets described above in Section IV.
**Fig. 1.**
General-Purpose Heat Source project schedule.

**Fig. 2.**
The radius and lower part of the side wall were drawn correctly, but a broken draw ring allowed wrinkling to occur in the upper side wall. Blank M-429. 1.5X.

**Fig. 3.**
The microstructure of the cup drawn from blank M-429 was free of recrystallized grains. 100 X.
Fig. 4.
The biaxial punch deformation resulted in failure of both unrecrystallized iridium disks. a) L252-1, control, and b) LR 280-2, laser etched.

Fig. 5.
The unrecrystallized iridium delaminated when it failed. a) L252-1, 150X; b) LR280-2, 700X, and c) LR280-2, 85 X.
Fig. 6.
The deeply etched base of the "2" perturbed the crack path in LR280-2. a) Optical macrograph, 10X, and b) scanning electron micrograph, 70X.

Fig. 7.
The punch-tested iridium disks failed at same strain levels. a) LR281-8, control, and b) L249-5, laser marked.
Fig. 8.
The grain structure of the recrystallized control sample, LR281-8, was nearly equiaxed. 100X.

Fig. 9.
The failure of LR281-8, the recrystallized, unmarked control sample, was primarily intergranular at the 965°C test temperature. 500X.
Fig. 10.
The microstructure of L249-5, the laser-marked iridium disk, was nearly equi-axed and its failure mode was intergranular. 100X.

Fig. 11.
The intergranular fracture mode of L249-5 is easily seen on the fracture surface. 500X.
Fig. 12.
Two cracks in L249-5 were associated with a "2" located away from the main failure. 3.2X.

Fig. 13.
The cracks associated with the laser etching were intergranular and the laser-induced pits had various depths. a) Base crack, 150X, and b) stem crack, 275X.
Fig. 14.
Photomacrographs of the plutonia sphere of MHF-428 illustrating a) high-density crust and b) low-density core, 10X.

Fig. 15.
Microstructures of the plutonia sphere of MHF-248. a) Crust and b) core. Note the grain boundary helium bubble strings in the crust sample, 250X.
Nonmetallic deposits at the entrance to the vent assembly of MHF-428 were identified as MgO, 25X.

Post-aging radiograph of the plutonia sphere of MHFT-74 showing extensive fracturing.

Photographs of a) the impact face and b) a profile view of MHFT-74. There were no fractures. 1X.
Fig. 19.
Microstructure of the weld bead of the iridium containment of MHFT-74. 40X.

Fig. 20.
Iridium deposit on the interior of the iridium shell of MHFT-74. 50X.

Fig. 21.
Iridium particles co-deposited with vapor transported plutonia in MHFT-74. 250X.
Fig. 22.
Photomacrographs of the plutonium sphere from MHFT-74 showing extensive fracturing. a) Impact face and b) core. 10X.

Fig. 23.
Photomicrograph of a region of extremely fine particles in the core sample of the plutonium sphere of MHFT-74. 250X.
Fig. 24.
Photomicrographs of the impacted plutonia sphere from MHFT-74 showing second phase impurity particles. 250X.

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<th>As-received</th>
<th>P-doped 500 h</th>
<th>P-doped 2000 h</th>
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<td>70 (S.D. 31)</td>
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<td>64</td>
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The specifications are: α Swipe, <220 counts/min
Helium Leak Rate, <1x10⁻⁶ cm³/s
Neutron Emission Rate, <6000 n/s·g²³⁸Pu
Calorimetry, 1.10 ± 0.03 W
### TABLE III

**NDT FOR ENCAPSULATED RHU PELLETS-2**

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<th>Fuel Pellet No.</th>
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<th>Helium Leak Rate (cm³/s)</th>
<th>Neutron Emission Rate (n/s-g²³⁸Pu)</th>
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*The specifications are:*  
- α Swipe, <220 counts/min  
- Helium Leak Rate, <1x10⁻⁶ cm³/s  
- Neutron Emission Rate, <6000 n/s-g²³⁸Pu  
- Calorimetry, 1.10 ± 0.03 W
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<tr>
<th>Fuel Pellet No.</th>
<th>Weight (g)</th>
<th>Capsule Body No.</th>
<th>α Swipe (Count/min)</th>
<th>Helium Leak Rate (cm³/s)</th>
<th>Neutron Emission Rate (n/s-g²³⁸Pu)</th>
<th>Calorimetry (W)</th>
<th>Comments</th>
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a The specifications are:
- α Swipe, <220 counts/min
- Helium Leak Rate, <1x10⁻⁶ cm³/s
- Neutron Emission Rate, <6000 n/s-g²³⁸Pu
- Calorimetry, 1.10 ± 0.03 W
### TABLE V
ENVIRONMENTAL TEST PELLET LOT ET2

<table>
<thead>
<tr>
<th>Feed Material</th>
<th>Hot Pressing Parameters</th>
<th>Post-Press Sintering</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;125-μm $^{238}$PuO$_2$ granules (Lots 39 and 57) seasoned at 1100°C (60 wt%) and 1600°C (40 wt%)</td>
<td>1530°C for 15 min at 19.5 MPa</td>
<td>6 h at 1000°C plus 6 h at 1527°C in Ar-H$_2$\textsuperscript{16}O</td>
<td>3 pellets, 15 W each</td>
</tr>
</tbody>
</table>

### Dimensions

<table>
<thead>
<tr>
<th>Condition</th>
<th>Diam (cm)</th>
<th>Length (cm)</th>
<th>Weight (g)</th>
<th>Density (% TD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Pressed</td>
<td>1.711 ± 0.001</td>
<td>1.692 ± 0.002</td>
<td>37.865 ± 0.007</td>
<td>85.5 ± 0.4</td>
</tr>
<tr>
<td>Sintered</td>
<td>1.698 ± 0.003</td>
<td>1.680 ± 0.003</td>
<td>38.129 ± 0.003</td>
<td>87.6 ± 0.2</td>
</tr>
</tbody>
</table>

### TABLE VI
ENVIRONMENTAL TEST PELLET LOT ET3

<table>
<thead>
<tr>
<th>Feed Material</th>
<th>Hot Pressing Parameters</th>
<th>Post-Press Sintering</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;125-μm $^{238}$PuO$_2$ granules Lots 38, 39, &amp; 60) seasoned at 1100°C (60 wt%) and 1600°C (40 wt%)</td>
<td>1530°C for 15 min at 19.5 MPa</td>
<td>6 h at 1000°C plus 6 h at 1527°C in Ar-H$_2$\textsuperscript{16}O</td>
<td>3 pellets, 15 W each</td>
</tr>
</tbody>
</table>

### Dimensions

<table>
<thead>
<tr>
<th>Condition</th>
<th>Diam (cm)</th>
<th>Length (cm)</th>
<th>Weight (g)</th>
<th>Density (% TD)</th>
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</thead>
<tbody>
<tr>
<td>As Pressed</td>
<td>1.706 ± 0.001</td>
<td>1.690 ± 0.002</td>
<td>37.902 ± 0.011</td>
<td>86.2 ± 0.1</td>
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<tr>
<td>Sintered</td>
<td>1.693 ± 0.002</td>
<td>1.677 ± 0.001</td>
<td>38.166 ± 0.011</td>
<td>88.2 ± 0.2</td>
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</tbody>
</table>
TABLE VII
ENVIRONMENTAL TEST PELLET ET4

Feed Material <125-um $^{238}\text{PuO}_2$ granules (Lots 38, 39, & 60) seasoned at 1100°C (60 wt%) and 1600°C (40 wt%)

Hot Pressing Parameters 1530°C for 15 min at 19.5 MPa

Post-Press Sintering 6 h at 1000°C plus 6 h at 1527°C in Ar-H$_2^{16}$O

Comments 3 pellets, 15 W each

Dimensions

<table>
<thead>
<tr>
<th>Condition</th>
<th>Diam (cm)</th>
<th>Length (cm)</th>
<th>Weight (g)</th>
<th>Density (% TD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Pressed</td>
<td>1.709±0.001</td>
<td>1.692±0.003</td>
<td>37.918±0.012</td>
<td>85.9±0.2</td>
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<tr>
<td>Sintered</td>
<td>1.701±0.006</td>
<td>1.684±0.005</td>
<td>38.183±0.012</td>
<td>87.1±1.1</td>
</tr>
</tbody>
</table>
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