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This monthly report is intended to inform the Space and Terrestrial Systems Division, DOE, and their contractors of significant technical highlights of technology and systems support programs in progress at Oak Ridge National Laboratory. A detailed technical presentation of information will be published in topical reports and open literature publications.

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SOLAR-POLAR MISSION MATERIALS SUPPORT

(Activity AE 15 15 20 0, WPAS 02313)

R. L. Heestand

The purpose of this task is to provide direct materials support on work specific to the General Purpose Heat Source (GPHS) design. The scope of this work includes (1) determining the impact properties and optimum weld parameters for the DOP-26 iridium alloy fuel cladding material, (2) thermal conductivity and emissivity data on cladding and thermal insulation materials, (3) fabrication and characterization of test samples of carbon-bonded carbon-fiber (CBCF) insulation developed by ORNL and Y-12 which may be used for GPHS, and (4) determination of creep properties of the 2219 aluminum alloy housing. Data on the impact properties and welding of iridium are integrated with complementary work reported in the Materials Technology section of this report.

CBCF-3 Insulation Fabrication (G. W. Brassell, R. L. Heestand)

No CBCF-3 parts were delivered during the past month. Out of eleven tubes submitted for re-inspection and eight tubes submitted for machining and inspection, none were acceptable due to being out of dimensional tolerances. The major dimension nonconformance was length with diameter as the second most frequent nonconformance. To remedy this problem, alternate machining sources are being investigated. These are primarily commercial machine shops. A considerable supply of as-deposited and carbonized tubes for machining are on hand. These include 18 blanks remaining from Y-12 and 27 blanks fabricated at ORNL. Machining of these parts will be initiated on selection of the machining source.

Pyrolytic Graphite (PG) Coating on FWPF (G. C. Wei)

Recent vibration tests of GPHS LMRE modules performed by GE have shown failures of CBCF-3 insulators. These results are contrary to previous results of vibration tests of simulated GPHS LMRE modules conducted by FI. These two sets of vibration tests were reviewed and possible solutions involving slight modifications of the LMRE design were assessed. A thin layer of pyrolytic graphite coating on the contact areas of the FWPF impact shell and Aeroshell can harden the contacting surfaces of the FWPF components and therefore improve the vibration survivability of the current GPHS LMRE design. Small pieces of FWPF for this PG coating experiment were received from GE on June 5, 1980. The PG coating furnace is being upgraded for this work.

QA Program of CBCF-3 Insulation (G. C. Wei and R. J. Beaver)

Specifications of machining and inspection of CBCF-3 parts for GPHS were completed. Procedures of chemical analyses including ash analysis and spark source mass spectroscopic analysis are being prepared. A QA program plan of CBCF-3 insulations for the GPHS systems is in process.

A subcontract with Purdue University to measure the thermal conductivity of CBCF-3 parts to establish a statistical-based thermophysical data base of CBCF-3 parts for GPHS safety verification tests, has been effective since May 15. However, specimens of the proper density prepared under the CBCF-3 fabrication task of this program were not available as of this reporting date.

Creep Properties of 2219 Aluminum Housing Materials (J. P. Hammond)

The purpose of this task is to develop creep design curves on the 2216-T6 aluminum forging and to aid GE, Valley Forge, in the design of the shell component of the RTG generator. The essential requirement of the shell will be to firmly retain the RTG generator under elastic stress during ground tests and storage in air (condition II), and subsequent flight in vacuum (condition I).

Since last reporting, long transverse (LT) test specimens were fabricated from the end cropping of the 2219-T6 forging and additional creep tests were placed in operation. General Electric, Valley Forge, communicated to us the agreed-on creep test matrix, which conforms generally with the test plans discussed in the last report. This matrix is shown in Table 1.

Table 1. Al 2219-T6 Alloy Creep Test Matrix

Test	Pretest Age	Test Temp (°F)	Stress (psi)	Time (h)	Orientation[a]
I	none	518	5000	10,000	LT
II	600 h @ 518°F 3500 psi	350	8000	10,000	LT
III Design Data	none	500	TBD(0.2% Creep)[b]	10,000	LT
IV Design Data	none	350	TBD(0.2% Creep)[b]	10,000	LT

[a]LT = long transverse.
[b]To be determined.

Tests I and II were initiated and three creep tests were started to provide Design Data III. Creep tests presently in operation on longitudinal (L) specimens at 350 and 500°F will be shortly terminated (after 1000 h) to initiate tests for Design Data IV.

Important Meetings

A. C. Schaffhauser and R. H. Cooper attended the GPHS Design Review Meeting conducted May 6, 1980, at DOE, Germantown.

FLIGHT SYSTEMS HARDWARE

(Activity AE 15 20 00 0, WPAS 01322)

D. E. Harasyn

The objective of this task is to supply Mound Facility with flight quality hardware components for use in the assembly of isotope heat sources. The major activity is on fabrication of iridium alloy forming blanks for isotope fuel capsules along with iridium foil for vents, decontamination covers, and weld shields. We have completed fabrication of iridium for the Galileo mission MHW heat source and have initiated fabrication 1255 iridium blanks and associated foil for Solar-Polar Mission General Purpose Heat Source for delivery in FY 1980 and FY 1981. A new task on fabrication of carbon-bonded carbon-fiber (CBCF) thermal insulation for Light Weight Radioisotope Heating Units (LWRHU) has also been initiated.

Iridium Blank Fabrication (D. E. Harasyn)

No forming blanks were shipped to MF this month while furnace and rolling mill modifications were made and the surface cracking problem described in last month's report was evaluated. Of the 355 GPHS blanks that were to have been shipped to MF in FY 1980, by the end of May (see April 1, 1980, GPHS Integrated Master Schedule), 245 have been shipped. Because evaluation of the first Ir sheet to be rolled with the modified equipment at Y-12 has not been completed, a firm schedule for additional blanks cannot be given at this time.

The modifications to equipment at Y-12 consisted of making an Inconel muffle for the furnace and installing wipers on the rolls. The Inconel muffle serves as a barrier to prevent contamination of the Ir sheet surface with firebrick. The wipers maintain the cleanliness of the rolls and distribute the lubricant. The first Ir sheet to be rolled after completing these modifications had excellent surface quality with no significant pits or surface inclusions. After the sheet was electro-etched, the surfaces appeared to have the same cracks as seen on blanks rolled on the Bliss mill (L264-L269). Such visual indications of surface cracks are not necessarily deleterious. We will be able to accurately evaluate the influence of these rolling modifications when the sheet has been blanked and the ultrasonic, liquid penetrant, and visual examinations are completed in late June.

The apparent "surface cracks" which have become prominent lately have always been present to some degree on all bare rolled Ir sheet. When reviewed at high magnification many have essentially no depth; and when viewed in cross section they again have no depth. Light surface treatments such as wire brushing remove many of them. They are not really cracks but perhaps only heavily etched grain boundaries and probably have no real consequence.

Some of the "surface cracks" are real cracks. These cracks trap liquid penetrant and show up as linear indications. Examination of some of these surface defects at high magnification indicated crack depths of around 0.01 mm (0.0004 in.). One crack viewed in cross section extended 0.03 mm (0.001 in.) below the surface and then ran parallel to the fibrous microstructure and the surface for 0.15 mm (0.006 in.). The large number of blanks with linear indications (14) in L264-L269 has forced us to resume bare rolling with the equipment at Y-12. Less than 1% of the blanks rolled with this equipment had linear indications. These real cracks do not appear to significantly influence the impact properties (see "Characterization of DOP-26 Ir-0.3% W Production Sheet"). However, they would require surface grinding to remove them. We reject or rework all blanks with penetrant indications, so those blanks with linear indications will not be shipped to MF.

The apparent surface cracks which can be seen at 10—30x may not be caused by the bare rolling process. The sheets L278, 280, and 281 were examined at 10—30x right after warm rolling and before bare rolling. Even in this condition the surface defects were visible so that they could be mapped out on paper. After bare rolling, flattening, and electroetching, the sheet was again visually examined. Areas of the sheet that had few surface defects after warm rolling had few defects after bare rolling, and areas with many defects after warm rolling had many defects after bare rolling.

The visually detected "surface cracks" may be simply heavily etched grain boundaries. The "cracks" form a cellular or grainy pattern on some blanks which allows one to estimate a grain size by the linear intercept method. This yields about 150 across the width of a sheet. A typical DOP-26 Ir—0.3% W casting has about 140 equiaxed grains across its width. In addition, the grainy pattern of "cracks" are slightly elongated in the major rolling direction as one would expect had they originated with the casting. We speculate at this time that the surface cracks in the rolled sheet surface are related to the grain boundaries of the casting.

We investigated the effects of rolling speed and sheet temperature on the frequency of the surface cracks. Sheets L276 and L277 were rolled under the conditions listed in Table 2, electroetched, and then examined. The rolling at 500°C was unacceptable because the sheet split severely. Rolling at high temperature and high speed produced the best results based on the number of liquid penetrant indications.

However, rolling speed and temperature may not be the most important variables. Table 3 shows a summary of the number of defective blanks from the first two lots rolled on the Bliss mill. Even though the two lots were rolled at the same temperature, 900°C, the same speed 4.0 m/min, and on the same mill, the number of linear penetrant indications were substantially lower in the second lot. We cannot identify at this time what the critical parameters are that control the surface cracks that trap liquid penetrant.

Iridium Foil Fabrication (D. E. Harasyn)

Approximately 190 cm^2 of undoped Ir—0.3% W 0.13-mm-thick foil was hand carried to MF this month. The foil is intended for experimental use to determine if the thorium and aluminum dopants in present production foil are influencing cracking in GPHS vent welds. The foil was rolled from reject WC and WG MHW blanks after sawing off the defective areas.

Table 2. Surface Cracking In Bare Rolled Ir Sheet Is Minimized By Rolling At High Speed And High Temperature

Sheet Preheat °C	Rolling Speed m/min	Penetrant Indications	Visual Crack Indications
1000°C	9.5	0	few
	4.0	5	many
500°C	9.5	0	moderate
	4.0	0	moderate

Table 3. Surface Cracking May Be Dependent On Variables Other Than Rolling Speed And Temperature

Defect Type	Number Of Defective Blanks	
	L264-L269 (Lot 1)	L270-L275 (Lot 2)
Ultrasonic	11	9
Penetrant-Linear	14	5
Visual	16	10

CBCF-3 Insulation for LWRHU (R. L. Heestand)

No CBCF-3 was fabricated for LWRHU since the design has not yet been selected.

MATERIALS TECHNOLOGY SUPPORT

(Activity AE 15 35 00 0, WPAS 01495)

C. T. Liu

The primary objective of this task is to characterize and improve the metallurgical and mechanical properties of noble base alloys, mainly DOP-26 (Ir–0.3% W doped with 60 ppm Th and 50 ppm Al), to meet the requirements of cladding material in radioisotope heat resources for the Galileo and Solar-Polar space missions. The current efforts are concentrated on four areas: (1) to characterize the impact properties of Ir alloys in the temperature range 800–1400°C, (2) to improve the low-temperature impact properties of welds in the DOP-26 alloy, (3) to identify the mechanism and sources that degrade the mechanical and metallurgical properties of doped Ir alloys under heat-source environments, and (4) to develop ductile high-temperature alloys acceptable for future flight missions.

Analysis of DOP-26 Exposed to Sulfur Vapor (C. L. White, H. Inouye and R. A. Padgett)

A specimen of DOP-26 type Ir–0.3% W (#800029) was given a 19 h vacuum anneal at 1500°C then exposed to ∿1.33 kPa sulfur vapor (probably S_2) for three days at 1000°C. This specimen was fractured in the AES vacuum system, and the resulting fracture surface was analyzed. Large concentrations of sulfur were observed on the fracture surface, however elemental mapping showed that the sulfur distribution was highly non-uniform. Sulfur (152 eV) to Ir (229 eV) peak height ratios as large as 8.6 were observed at certain locations on the fracture surface, while essentially no sulfur was detected at others. Inert ion sputtering (estimated to remove approximately two atom layers from the fracture surface) removed nearly all of the sulfur.

Weld Development (S. A. David and C. T. Liu)

At the request of C. O. Tarr of DOE, a trip to Mound Laboratory was made to meet with Ernie Johnson and other welding and processing personnel to discuss the welding problem related to the GPHS vent welds. As a result of this meeting the following actions were suggested.

1. use undoped Ir–0.3% W alloy for frit cover material,

2. a possible change in the frit cover design to reduce local strains around the welds,

3. continued line of investigations proposed by Mound, and

4. to initiate a study by ORNL to investigate the effect of grain size on weldability of DOP-26 alloy.

We are also continuing to evaluate the weldability of heat treated FS-3 foils to GPHS capsule sheet material (DOP-26). Two frit cover sheet materials were heat treated, one at 1300°C for 1 h and the other at 1500°C for 1 h and at 1900°C for 2 h to obtain different grain sizes in the material. Each one of these sheets was electron beam (EB) welded on to DOP-26 discs following a square geometry at four different focus conditions to study the weldability. Weldability of these frit cover materials has been found to be very good. No weld metal cracks were observed in any of these welds.

Analysis of Vent Welds in GPHS Capsules (C. L. White and R. A. Padgett)

Part of a GPHS capsule (LR-229-R3) containing a cracked vent weld was received from Mound Laboratory. This capsule was sectioned in an effort to obtain several specimens suitable for AES analysis. Only one specimen (#800028A) was obtained, however. The weld had apparently cracked completely through on all other specimens.

Specimen 800023A was fractured in the AES system, and grain boundaries in the weld region were analyzed. Small concentrations (on the order of 1 at. %) of Si and Sn were detected at the grain boundaries along with approximately the same level of the Th as is normally found in DOP-26 material. Scanning electron microscopy of the specimen, following AES analysis, failed to reveal any unusual precipitate structure at the weld metal grain boundaries.

Characterization of DOP-26 Ir–0.3% W Production Sheet (D. E. Harasyn and C. T. Liu)

The purpose of this subtask is to characterize production lots of Ir–0.3% W sheet. Primarily this involves reporting the results of uniaxial impact tests performed on each production lot. A production lot is normally 48 blanks fabricated from six ingots.

The samples prepared from different lots with surface defects were annealed for 19 h at 1500°C and then impacted at the GPHS conditions (980°C and 200 fps). The impact data in Table 4 show that the impact properties of recrystallized DOP-26 Ir–0.3% W samples are not sensitive to surface defects. The samples from the sheet L-216 contained surface inclusions in the gage lengths. One measured about 100 μ in diameter and the other about 30 μ. The inclusions were examined with an x-ray energy dispersive analyzer on an SEM and found to contain Al, Ca, Ba, Fe, Ti, Zn and S. It cannot be said with certainty that these inclusions were firebrick; however, it is not unusual for firebrick furnace linings to become heavily contaminated by the materials placed in the furnace. Re-examination of the samples after a 19 h anneal at 1500°C showed no trace of the inclusions. The same areas were analyzed under the SEM and found to be free of detectable contamination. Apparently most of the contaminants had volatilized during the heat treatment. The impact tests showed that the inclusions did not influence the ductility of DOP-26 alloy.

Table 4. Tensile Impact Data on DOP-26 Production Sheets[a]

Sheet No.	Uniform Elongation	Reduction of Area	Fracture Mode[b]	Remarks
WK-12	16.4	25	TF+GBS	
L216 (A1)	18.7	36	TF+GBS	Surface inclusions in gage length
L216 (A2)	19.8	37	TF+GBS	Surface inclusions in gage length
L264	17.8	26	TF+GBS	C-brale indentions .07 mm (.0028 in.) deep, 1 cm apart in gage length
L268-5	15.5	30	TF+GBS	Linear liquid penetrant indications in gage length
L268-5	14.7	25	TF+GBS	
L272 (A)	7.4	9	GBS	Contaminated with 100 μg/g Zr

[a]Samples annealed 19 h at 1500°C, impacted at 61 m/s at 980°C.

[b]TF = transgranular fracture, GBS = grain boundary separation.

The sample from L-264 contained two C-brale hardness indentions located 1 cm apart in the gage length. The indentions measured .07 mm (.0028 in.) deep by .38 mm (.015 in.) diameter at the surface. The indentions did not influence the impact ductility as indicated in Table 4. Because the deepest pits in production hardware made after Inconel muffles have been used for the bare rolling process, are about .03 mm deep, the impact data indicate that pitting in Ir PICS will not be a problem as far as impact ductility is concerned.

The two impact samples from L-268-5 contained linear liquid penetrant indications in the gage lengths. These indications presumably resulted from shallow surface cracks which have been observed in some metallography samples of production hardware. The cracks typically run .03 mm below and parallel to the surface for about .15 mm. As compared with other surface defects, these appear not to influence the impact ductility.

The sheet L-272 was contaminated with 100 μg/g Zr. In contrast to similar samples impacted under MHW conditions, this sample showed a distinct decrease in ductility and a change in fracture mode to completely grain boundary separation. We intend to check the result with another impact, but at this time it appears that this high level of Zr cannot be tolerated in GPHS.

Mechanical Properties of Pt—30 Rh (J. R. Keiser)

In order to collect needed mechanical property data for the LWRHU design effort, both high temperature slow strain rate tests and low temperature high strain rate tests have been run on Pt—30 Rh. The high temperature tensile tests were run to determine if the alloy is strong enough to contain the pressure of internally produced helium during a heat pulse. Samples annealed 1 h at 1000°C then tested at a strain rate of 0.2 min^{-1} gave the following results.

Test Temperature (°C)	Ultimate Tensile Strength MPa	(psi)	0.2 Offset Yield Strength MPa	(psi)	Total Elongation (%)
1400	56.3	8172	43.3	6280	26.6
1515	40.2	5829	32.7	4739	70.0

The low temperature tests were run to determine the ductility of Pt—30 Rh in the event of a high velocity impact. Tests run at 100°C on specimens which had been annealed 1 h at 1200°C gave the following results:

Strain Rate m/s	ft/s	Total Elongation (%)
111.3	365	34.4
114.5	375.5	39.7

ISOTOPE FUEL DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 02314)

R. S. Crouse

$^{90}SrF_2$ Compatibility (R. S. Crouse)

Metallographic examination of the 30,000 h $^{90}SrF_2$ compatibility samples is complete. The tests originally were to be run at 600, 800, and 1000°C but we only received those run at 600 and 800°C.

The general pattern established in the shorter term tests carried through these tests. TZM was far less heavily attacked than the Hastelloy C-276 and the Haynes 25. Maximum depth and severity of attack was seen in the Hastelloy C-276 with Haynes 25 showing a less severe attack but far worse than TZM. The table below gives the maximum attack found in each alloy at each temperature.

Alloy	Depth of Attack (μm)		Comments
	600°C	800°C	
TZM	nil	40	Only surface effect.
C-276	210	440	Attack zone spongy. Mixture of metallic and nonmetallic.
Haynes 25	70	300	Attack zone spongy. Subsurface voids to 250 μm below attack.

It was notable that attack varied greatly from one sample to another at the same temperature. This was the case for each sample except for Haynes 25 at 600°C. An example of this variation was Hastelloy C-276 at 800°C. One sample showed about 20% deeper attach with noticeably greater severity within the attack zone. No reasons for this variation are readily discernible.

Microprobe analyses will be started on these samples in June.

TERRESTRIAL RADIOISOTOPE APPLICATION DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 01367)

F. N. Case, K. W. Haff, and F. J. Schultz

Cesium-137 Low Solubility Compounds

Sectioning of the ^{137}Cs pollucite pellets was completed this month. Two pieces from each of four test pellets (total of eight pieces) were transferred to the HRLEL for characterization studies. One piece was obtained from the approximate center of the pellet, while the other was an adjoining piece containing an outside surface of the original pellet.

Optical microscopy studies of the eight pollucite pellet pieces were completed. Pictures (magnifications ranging from 50X to 750X) obtained of the pellet sections using a metallograph reveal a two-phase system having a moderate porosity. The predominant phase is gray in color and contains white flecks interspersed throughout (the second phase). The black pores and white flecks become more numerous as one moves away from the outside sample of the pellet towards the center. This indicates a density gradient exists from the center surface to the more porous interior. The white flecks observed throughout the gray matrix probably contain a larger percentage of aluminum and silicon. They appear white due to the higher reflectivity of the aluminum and silicon. The pores were probably formed as a result of outgassing and/or inhomogeneous melting of the pollucite powder during the hot pressing procedure.

The pollucite pellet calorimeter water lines and thermocouple line were disconnected and the calorimeter moved to a storage area in the hot cell. This is in preparation for transfer of the Soxhlet leaching apparatus and associated glassware into the hot cell early next month.

A discussion with the staff at the HRLEL concerning scanning electron microscopy and electron microprobe studies of the ^{137}Cs pollucite pellets was held this month. The operator of the SEM was instructed to examine the vertical and horizontal axes of the pellets for a cesium gradient. Also, he should determine if there exists a difference in cesium, aluminum, and silicon content between the two phases. These studies will begin in early June.

Krypton-85 Light Source Development

The eight lights required for the July tests at Andrews Air Force Base were fabricated this month. Design of the inner shipping container was completed and fabrication begun. The tests are scheduled for the week of July 14-18, 1980.

BENEFICIAL APPLICATIONS OF RADIATION TECHNOLOGY

(Activity AE 15 35 00 0, WPAS BART001)

C. S. Sims

Preparations to perform radiation dose measurements at the Sandia Irradiator for Dried Sewage Solids have been initiated. Current plans call for three different types of dosimeters (lithium tetraborate thermoluminescent, ceric sulfate, and a plastic yet to be specified) to be used for independent checks. H. W. Dickson of the Health and Safety Research Division will direct the effort targeted for August, 1980.

Personnel associated with the BART Program traveled to Atlanta, Chicago, Los Angeles, and New York to attend Environmental Protection Agency public meetings on a draft regulation for distribution and marketing of sludge-derived fertilizers and soil conditions (i.e., 40 CFR 258). Public comments ranged from "too strict" to "not strict enough." In general, the public voiced more concern about heavy metals than about pathogens.

Work continued on the development of the program plan and the organization of the BART Program for FY 1980 and beyond.

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