DATE ISSUED FEB 11 1981
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ORNL/CF-81/29

810206

DATE: February 5, 1981

SUBJECT: Technical Highlights of Space and Terrestrial Systems Programs at Oak Ridge National Laboratory for November 1980

TO: Distribution

FROM: R. H. Cooper

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

This task provides the fabrication development, characterization, and design verification of materials for the General Purpose Heat Source (GPHS) and Thermoelectric Converter for the 1983 Solar-Polar Space Mission.

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

The purpose of this task is to develop creep testing curves on the 2219 aluminum forging used to retain the RTG generator under elastic stress during ground test, storage, and flight. Creep tests are underway, however, there is no data to present this period.

FLIGHT SYSTEMS HARDWARE

(Activity AE 15 20 00 0 WPAS 01322)

R. L. Beatty and R. L. Heestand

The objective of this task is to supply Mound Facility with flight quality carbon-bonded carbon-fiber insulators and iridium hardware components for use in the assembly of isotope heat sources. The major iridium activities are associated with fabrication of iridium alloy forming blanks for isotope fuel capsules and the fabrication of iridium foil for vents, decontamination covers, and weld shields. This task also includes management of the precious metal inventory and iridium refining activities for space systems and the coordination of iridium transfers between contractors involved in the General Purpose Heat Source Program.

In addition ORNL is fabricating cylindrical insulator sleeves and end discs from a carbon-bonded carbon-fiber material. Techniques to fabricate this light-weight high-temperature insulating material in the components needed for the General Purpose Heat Source were developed at ORNL in FY-1980.

Iridium Forming Blank Fabrication (R. L. Heestand)

Thirty-seven ground forming blanks were shipped to Mound Facility early in the month of November. The discs consisted primarily of rework and recycle of "L" material and "M" material which had been remelted to raise the impact ductility. The improved impact ductility of the eight times electron "M" material was reported previously.[1] Although the impact ductility of the "M" material is improved by an additional four electron beam melts, these additional melting double the material loss typically associated with electron beam melting.

Dye penetrant inspection results of cups formed at Mound Facility from the first shipment of ground blanks was received. This information indicated that cracking was observed in the cup radius which was related to the grind marks and possibly related to the rolling direction. With the current ORNL procedure, the grinding and rolling directions coincide, a convention established during MHW fabrication. Because of the cracking seen in cups formed from grounded blanks, a decision was made at the November Heat Source meeting to rework the ground surface of ORNL blanks by lapping. In addition, all blanks were to be stress relieved after grinding and lapping. Six delamination reject blanks were used in a lapping experiment to establish a lapping procedure necessary to eliminate the grind marks. As the majority of discs were already ground to the midrange of the tolerance, it was found only one side of each blank could be lapped and hold the blanks within thickness tolerance.

A summary of the iridium blank and foil fabrication status by ingot lot number (Table 1) and indicates that ingot numbers for the 37 blanks shipped this month. This data also indicates a possible December shipment of possibly 82 blanks.

Table 1. Iridium Blank and Foil Fabrication Status by Lot Number[a]

Material Identification	Status	Expected Blanks
LR306-310, M422-MR424	Shipped	37
M428-433	Quality Assurance Record audit	22
MER1-3	NDT	18
N510-515	Arc casting	36
LR311-312	Dropcast — on hold for experimental use	12
N501-509, LR300	Grinding	60
MD4-2, 5, 6	Foil rolling	Foil
"O" powder	EB melting (10 troy oz)	Foil
"P" powder	Compacting (400 troy oz)	

[a]From November 14, 1980, Biweekly Status Report.

Iridium Management (W. O. Graves)

The 37 kg of iridium scrap was shipped to Engelhard for refining. Assay samples are expected to be available from Engelhard by the end of December. In addition the requested transfer of 25 troy ounces of iridium from the Engelhard pool account to ORNL was completed.

CBCF-3 Insulation Fabrication and Characterization (J M Robbins)

The new liquid-seal vacuum pump for dewatering CBCF moldings was received and installed. The pump was manufactured by Beach-Russ Company and has a pumping capacity of 125 CFM. Two billet runs (C-33 and C-34) and one plate run (P-10) have been made with the new pump. There has been

some question about the possibility of fines carbon fibers segregating toward the bottom of the fiber drum, thus, increasing density as fibers nearer the bottom of the drum are used. Billet run C-33 was made using fibers from the nearly empty original drum, and C-34 was made using fibers from the top of a new drum. Densities for runs C-33 and C-34 were 0.23 and 0.20 Mg/m^3, respectively. There is an indication that some size distribution difference of the fibers does exist between the top and bottom of the drum and that this size difference affects the density of the molded part. Measurements of selected samples of fibers are underway to refute or confirm this supposition.

A total of 23 billets from runs C-29, C-33, and C-34 have been submitted for finished machining to meet the immediate requirement of 16 CBCF insulation sets. The matching endcaps or discs are being machined from plate No. 8.

An inventory of the carbon fiber used in CBCF insulation fabrication was performed. Based on current usage, the fiber on hand will produce the following parts:

90% yield - 365 sets
80% yield - 325 sets
70% yield - 280 sets
60% yield - 240 sets

This analysis indicates that a 90% overall yield will be required to meet the 361 part requirement currently identified in the Mound Facility integrated schedule. We believe that it is highly unlikely that an overall process yield of 90% will be realized. In view of this uncertainty, an additional batch of carbon fibers should be qualified.

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 focused in four areas: (1) to provide continuing technical support of the ORNL iridium production activity; (2) to coordinate and interface ORNL materials technology activities with the needs of LANSL, MF, SRP/L and the systems integration contractor; (3) to determine the effect of impurities and environment on the impact properties of the DOP-26 iridium alloy; and (4) to characterize the hot cracking, fusion zone structure, and impact properties of arc welded and laser welded DOP-26 iridium alloy. With formation of the Iridium Task Force, there has been a major shift in emphasis to the first two efforts described above.

Characterization of Defects on Forming Cups (R. L. Heestand and H. Inouye)

In the study of radius cracking during forming, areas of nonuniform deformation have been associated with the crack areas. In a recent iridium task force meeting it was pointed out that the current rolling schedule may not be producing an optimum structure for forming. To determine whether the current rolling schedule is producing a suitable structure, a rolling experiment was designed and initiated which consists of sectioning the ingot at critical stages in the reduction and evaluating the microstructure resulting from varying recrystallization temperatures.

Iridium Forming Experiments (R. L. Heestand)

A series of forming experiments were carried out to evaluate the effects of surface condition and stress relief temperature on defect location and frequency. Table 2 lists the conditions and defects resulting from the forming. The majority of the experiments were conducted using blanks rejected for delaminations or minor surface defects. Experiments 1 through 3 were conducted to determine the effect of increasing temperature and the maximum forming temperature possible utilizing existing ORNL equipment. As was anticipated the higher temperatures soften the tooling, as a result, two additional sets of tools were fabricated from H-13 steel. In addition, a high temperature die steel was also ordered to fabricate two sets of dies capable of operating at 700°C.

Experiments 4 through 6 were conducted to determine the effects of oxidation on removing stress risers in the surface and the effect of stress relief. This test had fewer defects than any conducted but requires addtional test for confirmation of the data. Electropolishing should give similar results.

Table 2. Iridium Forming Experiments

Experiment No.	Disc No.	Surface[a]	Stress Relief (T°C)	Forming (T°C)	Defects after Forming[b]
1	MR426-1	G	—	580	1 ODR - 1 IDW
2	MR426-2	G	—	650	2 ODR - 2 IDW
3	MR426-3	G	—	650	2 ODR - 1 IDR
4	M402-7	BR	1000 Air	600	3 ODR
5	M428-3	G	1000 Air	600	1 ODW - 1 IDW
6	M405-7	G	1000 Air	600	None
7	M404-4	G-L	—	600	6 ODR - 1 IDR
8	M404-6	G-L	—	600	4 ODR - 4 IDR - 3 ODW
9	M405-3	G-L	—	600	4 ODR - 4 IDR
10	LR300-3	G 45°	970	600	2 ODR - 1 IDR
11	LR300-6	G 45°	970	600	2 ODR - 1 IDR
12	M402-2	G-L	970	600	2 ODR - 1 IDR
13	M403-5	G-L	970	600	3 ODR - 2 IDR
14	MER1-6	G-L	970	600	1 ODR - 2 IDR
15	MERR3-6	G-L	970	600	2 ODR
16	MR432-1	G-L	970	600	None
17	M404-7	G-L	1060	600	6 ODR - 2 IDR
18	M405-4	G-L	1060	600	2 ODR - 2 IDR
19	M405-5	G-L	1060	600	1 ODR

[a]BR = bare rolled, G = ground, L = lapped.

[b]OD = outside diameter, ID = inside diameter, W = wall, R = radius.

Experiments 7 through 9 show the results of forming a ground and lapped finish without stress relief. This gave an excessive number of defects. In experiments 12 through 16 it is shown that stress relieving the ground and lapped surface decreases the frequency of defects but does not eliminate the problem. Experiments 17 through 19 were conducted on ground and lapped blanks stress relieved with a small amount of recrystallization which apparently increases the defect frequency.

Experiments 10 and 11 were conducted to determine the effect of grinding at 45° to the rolling direction which essentially made the defects more random.

Specimen LR280-8 was originally formed at ORNL with no defects detected by the ORNL dye penetrant method. This specimen was transferred to MF where several defects were found using the MF technique. This specimen has been returned to ORNL for further dye penetrant sensitivity studies and metallographic examination of one selected defect.

Effect of Frit-Vent and Decon-Cover Welds on Impact Properties of DOP-26 Base Metals (C. T. Liu)

The new frit-vent and decon-cover welds performed at MF have shown a deep penetration into the base metal of the GPHS capsules. To study the effect of this penetration on impact properties of the base metal, DOP-26 disks were annealed 1 h/1300°C plus 1 h/1500°C and welded with frit-vent and decon-cover sheets at MF. Tensile specimens were then "eloxed" from the welded disks with the welds located in the gage section. Table 3 shows the impact properties of the welded specimens tested at 980°C and 61 m/s (200 fps). The as-welded specimen had an impact ductility of 13.0%. A post-weld heat treatment of 18 h at 1500°C increases the impact ductility to 14.1–15.7%. As compared with the impact ductility of unwelded specimens (Table 3), the frit-vent and decon-cover welds only cause a small reduction in the impact ductility of the DOP-26 base metal.

Table 3. Effect of Frit-Vent and Decon-Cover Welds on Impact Properties of DOP-26 Tested at 980°C and 61 m/s

Weld and Heat Treatment Condition	Impact Ductility (%)
As-Welded[a]	13.0
As-Weld[a] + 18 h/1500°C	15.7
As-Weld[a] + 18 h/1500°C	14.1
Base Metal + 19 h/1500°C	17.9 ± 3.2

[a]Specimens were annealed 1 h at 1300°C + 1 h at 1500°C prior to vent and decon-cover welding.

Microanalysis Studies (C. L. White and R. A. Padgett)

The laboratory has recently purchased a model 590 Scanning Auger Microprobe (commonly called "Super SAM") from the Physical Electronics Division of Perkin Elmer Corporation. This facility will be available for use on space and terrestrial activities. This facility permits specimens to be fractured under ultra-high-vacuum and the resulting fracture surfaces to be analyzed using AES at spatial resolutions on the order of 2000 Å. Auger analysis is performed using a cylindrical mirror analyzer (CMA) with a coaxial electron gun. This arrangement minimizes shadowing effects on rough fracture surfaces. Normal incidence sputter-ion-etching can also be performed in the same analysis chamber.

This facility, along with computer assisted data acquisition and processing, has provided a significant increase in our capability to study solute segregation to grain boundaries and other internal interfaces. The improved spatial resolution (2000 Å versus ~ 5 μm in our older facility) allows individual grains and precipitates to be selected for analysis, even on fairly small grained materials. Computer assisted data acquisition helps to minimize set up time for repetitive analyses, and allows different sets of data to be easily displayed in comparable formats.

Unfortunately, Super SAM does not currently have a vacuum transfer lock, which means that the time required to insert a specimen into the analysis chamber and obtain a vacuum of $\sim 10^{-8}$ Pa has increased (from ~12 h for our older facility to ~48 h with Super SAM). The specimen geometry in Super SAM is also more demanding, requiring special adaptors to be made for iridium alloy sheet specimens. These adaptors are currently being fabricated, and should be completed soon. We expect that the increased capabilities of this facility will greatly benefit the Space and Terrestrial Systems Programs.

TERRESTRIAL RADIOISOTOPE APPLICATION DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 01367)

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

Cesium-137 Low Solubility Compounds

Leaching of the sectioned and unsectioned fully loaded ^{137}Cs aluminosilicate pellets, as well as the tracer level irradiated pellets, is in progress. Table 1 shows the total elapsed time each pellet specimen has been leached.

Table 1. Total Elapsed Leaching Period of Test Pellet Specimens

Pellet specimen	Total elapsed leaching period (days)
Quasi-static,[a] sectioned, fully loaded ^{137}Cs aluminosilicate pellets	166
Static,[b] unsectioned, fully loaded ^{137}Cs aluminosilicate pellets	126
Quasi-static, sectioned, tracer-level ^{137}Cs aluminosilicate pellets	126

[a]Soxhlet extractor leaching.

[b]Simple immersion in distilled water.

Due to a malfunction of the gamma-ray spectrometer system the leachant samples were not assayed for their ^{137}Cs content this month. When the system is reinstated the samples will be assayed and the results reported.

The first draft of the technology transfer document for the ^{137}Cs source compound program entitled "Preparation and Characterization of Cesium-137 Aluminosilicate Pellets for Radioactive Source Applications", is being reviewed and edited.

Krypton-85 Light Source Development

Two quartz krypton light source containers were pressure tested this month. The conical-spiral quartz tube assembly was filled with argon gas and the pressure increased until failure occurred at 750 psig. Upon examination of the tube assembly after the test it was observed that the assembly had slipped out of its holder during the test. This was the direct cause of failure of the unit rather than a pressure failure. The second light source assembly tested was a one inch diameter right cylindrical tube within another tube. The unit was pressurized within the annulus between the tubes which would contain the krypton gas in use. The source assembly failed after 45 seconds at 500 psig.

The wavelength spectrum of a blue light source was measured over the range of 250 to 800 nanometers. The source was nearly monochromatic with emission at 460 nanometers.

The hollow frustrum of cones phosphor support light source is being fabricated. This phosphor support system will be utilized in the conical spiral tube geometry. This configuration offers a 14% increase in primary (non-reflected) light emission surface.

REFERENCES

1. C. T. Liu, Technical Highlights of Space and Terrestrial Systems Program at Oak Ridge National Laboratory for October 1980, p. 14.

MEETINGS

A. C. Schaffhauser, H. Inouye, C. R. Bieber, and R. H. Cooper attended an Iridium Task Force Meeting at Mound Facility, November 5, 6, 1980.

H. Inouye, H. Kling, and R. L. Heestand held an Iridium Task Force Defects Subgroup Meeting at ORNL, November 12, 1980.

R. L. Heestand, C. R. Bieber, and E. W. Johnson held and Iridium Task Force Forming Subgroup Meeting at ORNL, November 14, 1980.

R. H. Cooper presented a review of the Space and Terrestrial System Program at the Metals and Ceramics Division Information Meeting, November 12, 1980.

R. H. Cooper hosted a meeting of General Electric and Mound Facility personnel on CBCF-3 Requirements at ORNL, November 14, 1980.

R. H. Cooper, A. C. Schaffhauser, and C. R. Bieber attended the GPHS Status Meeting at Mound Facility, November 20, 1980.

R. J. Beaver hosted an Iridium Task Force Quality Assurance Review at ORNL, November 19, 1980.

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