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SUBJECT: Technical Highlights of Isotopic Power Materials Development at Oak Ridge National Laboratory for January 1980

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FROM: A. C. Schaffhauser

This monthly report is intended to inform the Division of Advanced Nuclear Systems and Projects, 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.

[redacted]

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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, and (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. 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. C. Wei, G. W. Brassell, L. Kovach, C. M. Stogsdill, C. D. Montgomery, and F. D. Bender)

Two sets (one set refers to one sleeve and two disks for one GIS) of finished CBCF-3 parts were delivered to FI for their vibration tests of the most recent GPHS module design (LMRE) involving CBCF-3. In addition, two sets (one set refers to one body and two caps) of finished CBCF-3 parts plus PG buttons and graphnol graphite rings which were machined according to a previous GPHS module design were sent to FI for possible uses in their dynamic tests of the most recent design.

Machining, X-ray radiography, and inspection of three plus spare sets of CBCF-3 parts for LASL impact tests were started. The finished parts are expected to be completed and shipped on February 8, 1980 to LASL for impact tests of the most recent GPHS module design using CBCF-3 (LMRE).

Based on discussions with FI, we agreed that the dimensional tolerance should be ± 0.076 mm (±0.003 in.) for finished CBCF-3 parts. We expect a 75% yield in the preproduction CBCF-3 parts for such tolerance.

One preproduction run using the multiple-molding facility (9 molds simultaneously) was completed. There appears to be no cracks in the carbonized CBCF-3 cups. The cracking problem encountered previously apparently was solved by proper control of the drying process. We plan to examine these nine parts with X-ray radiography to confirm the uniformity and defect-free state of the carbonized CBCF-3 cups. Subsequently, these nine cups will be machined, inspected, and shipped to LASL for safety tests.

Since the most recent GPHS module design (LMRE) uses flat disks at the ends of each GIS, large flat disks of CBCF-3 are desired for machining into such end disks. Fabrication of three 203-mm diam by 25-mm thick CBCF-3 disks was completed. We will evaluate their densities and compressive strengths. Adjustment in the material formulation and process variables of these large disks might be required in order to produce flat disks having the same density as the cups (0.20E3 kg/m^3).

Cost estimates of fabrication and QA of 22 sets plus spares for LASL safety-related tests (April) and 12 sets plus spares for Mound Lab process certification (June) were completed and forwarded to DOE Headquarters.

CBCF-3 Insulation Characterization (G. C. Wei)

Chemical analysis of a typical as-made CBCF-3 part sent to LASL or FI was completed. The results are shown in Table 1. Note that the trace impurities listed in Table 1 refer to as-made CBCF-3 and a proper outgassing treatment (1500°C-36 h-1.0E-6 torr) can reduce the trace impurity concentrations considerably. The chemical analysis of an outgassed CBCF-3 cup is in progress. The impurity concentrations shown in Table 1 are somewhat higher than those (Table 2) of as-made CBCF-3 disks manufactured one year ago for the Selenide Isotope Generator Program. The difference in the impurity concentrations probably is due to the different batches of carbon fibers used in CBCF-3 cups (Am. Enka AEPC-2) and CBCF-3 disks (Am. Enka AEPC-1). Samples of carbon fibers (AEPC-2) and phenolic resins were submitted to Analytical Chemistry for chemical analysis.

Thermal conductivity and compressive strength specimens were machined from an aged (1350°C/1100 h), outgassed (1500°C-36 h-1.0E-6 torr) CBCF-3 plate sample. However, thermal conductivity testing was on hold because our contract fund at Purdue University where the thermal tests would be performed was exhausted after contact conductance measurements described in the next section. Room temperature compressive strength (∿1.4 MPa or ∿200 psi) of the aged outgassed specimens indicated no significant aging effect on the compressive strength. Measurements of elastic modulus and compressive strength at high temperatures were also on hold due to the limitation of available operating funds.

Analysis of Graphite Compliance Pad Materials (G. C. Wei)

Recently, some members of the GPHS community expressed their concern about the use of grafoil in GPHS from the viewpoint of its high impurity concentrations which could be detrimental to iridium fuel cladding. Consequently, we analyzed the trace impurities of one sample of outgassed (1500°C-36 h-1.0E-6 torr) grafoil. The results are presented in Table 3. The not-so-low trace impurities shown in Table 2 confirmed the concern about impurities in grafoil. However, it should

be noted that a higher temperature and longer time outgassing treatment may reduce the impurity content in grafoil. We also assessed alternate materials for use as compliance pad. These include woven fabric made from Thornel 50 graphite fiber (Prodesco fabric No. 1099) and graphite cloth (UCC WCG). However, the chemical purity of these two commercial products has to be confirmed.

Table 1. Chemical Analysis of As-Made CBCF-3 Cup

Element	Concentration (wt ppm)	Element	Concentration (wt ppm)	Element	Concentration (wt ppm)
Ag	<5	Mo	<6	U	50
Al	50	Na	3	V	<5
As	<1	Nb	<3	W	<40
B	7	Ni	4	Zn	5
Ba	10	P	20	Zr	<5
Bi	<5	Pb	<5	Se	<4
Ca	300	Pd	<10	I	<3
Cd	<10	Rb	<1	F	<2
Co	<1	Rh	<3	Br	<3
Cr	20	Sb	<5	Cl	20
Cs	<0.3	Sc	<1	S	50
Cu	<2	Si	70	Sc	<1
Fe	300	Sn	<10	Y	<2
Ga	<2	Sr	8	La	<3
Ge	<3	Ta	50	Ce	<3
Hf	<10	Te	<10	Pr	<3
K	4	Th	<10	Nd	<20
Mg	<30	Ti	<20		
Mn	2				

Table 2. Chemical Analysis of As-Made CBCF3 Disks Made in 1978 and 1979

Element	Concentration (wt ppm)		
	CBCF3 (TR4)	CBCF3 (TR5)	CBCF3 (TR6)
Ag	<3	<3	<5
Al	3	10	50
As	<3	<3	<3
B	1	3	3
Ba	<3	<3	<3
Be	<0.3	<0.3	<0.3
Bi	<5	<5	<5
Ca	100	100	100
Cd	<3	<3	<3
Co	<1	1	<1
Cr	1	3	3
Cs	<3	<3	<3
Cu	5	15	<1
Fe	30	100	100
Ga	<3	<3	<3
Ge	<3	<3	<3
Hf	<10	<10	<10
Hg	<5	<5	<5
K	100	100	100
Li	<0.1	<0.1	<0.1
Mg	20	<50	20
Mn	3	3	<1
Mo	<3	<3	<3
Na	5	5	5
Ni	5	5	5
P	20	20	5
Pb	<5	<5	<5
Pd	<3	<3	<3
Rb	<3	<3	<3
Rh	<3	<3	<3
Ru	<3	<3	<3
Sb	<3	<3	<3
Sc	<1	<1	<1
Si	<30	<30	30
Sn	<3	<3	<3
Sr	<3	<3	<3
Ta	<5	<5	<5
Te	<3	<3	<3
Th	<5	<5	<5
Ti	<50	<50	<50
Tl	<5	<5	<5

Table 3. Chemical Analysis of Outgassed Grafoil Concentration
Unit in wt ppm

Element	Concentration (wt ppm)	Element	Concentration (wt ppm)	Element	Concentration (wt ppm)
Ag	<1	In	<3	Sc	≤2
Al	5	Ir		Si	300
As	<3	K	7	Sn	<3
Au		Li	5	Sr	<1
B	≤1	Mg	2	Ta	<5
Ba	2	Mn	5	Te	<3
Be	<.3	Mo	<3	Th	<5
Bi	<5	Na	<2	Ti	≤3
Ca	30	Nb	<3	Tl	<5
Cd	<3	Ni	4	U	<5
Co	1	Os		V	7
Cr	10	P	3	W	<10
Cs	<.3	Pb	<5	Zn	3
Cu	2	Pd	<3	Zr	3
Fe	200	Pt		S	500
Ga	≤3	Rb	≤1	Cl	30
Ge	<3	Re		I	<3
Hf	<10	Rh	<3	Br	<3
Hg		Ru	<3	Se	<3
Sb	<3	F	<3	Y	<3
La	<3	Ce	1	Pr	<1
Nd	<5				

Bonding of GPHS Components (G. C. Wei)

In preliminary experiments, we have bonded CBCF-3 to FWPF 3D C-C by using phenolic resin and grafoil to FWPF 3D C-C by using Dylon graphite cement. The phenolic resin is cured at 100°C for 24 h and the Dylon graphite cement at 100°C for 24 h. Both bonds were carbonized at 1350°C for 3 h in vacuum. The bonds so formed remained integral after normal handling. Samples of Dylon cement and carbonized phenolic resin were submitted for chemical analysis. We also plan to determine the penetration depth of the phenolic resin bond into CBCF-3.

Contact Conductance (G. C. Wei)

Contact conductance measurements of several material systems were performed by Dr. Ray Taylor of Purdue University using transient method. The results are shown in Table 4. The room temperature contact conductance (0.016 $W/cm^2 \cdot K$) of CBCF-3 to FWPF 3D C-C in vacuum is lower than the assumed value (0.14 $W/cm^2 \cdot K$) used by the GPHS community in the past but the room temperature contact conductance (1.3 $w/cm^2 \cdot K$) of FWPF 3D C-C to FWPF 3D C-C in vacuum is higher than the assumed. We also initiated an in-house effort on the contact conductance measurement using steady state method performed by D. L. McElroy and R. K. Williams.

FWPF 3D C-C Characterization (G. C. Wei)

We calculated the emissivity of FWPF 3D C-C along the Z-axis. It is $E = A + BT + CT^2$ where A = 0.59326, B = 1.62596E-4, C = 1.47196E-8, and T is temperature in kelvin.

Important Meetings and Correspondence

1. R. L. Heestand, G. C. Wei, and G. W. Brassell visited Mound Lab to discuss specifications of CBCF-3 insulation for GPHS on February 5, 1980.

Table 4. Preliminary Data of Contact Conductance ($W/cm^2 \cdot K$)

Material System	Temperature (°C)	Pressure (psi)	Vacuum	0.1 atm Air
FWPF vs FWPF	RT	5	1.3	1.58
		75	5.3	6.3
		175	22	22
	300	5	*a*	*a*
		75	*a*	3.7
		175	*a*	*a*
PG vs FWPF (Z11)	RT	5	Similar to (~15%)	0.85
		75	0.1 atmair	73
		175	Data	*b*
PG vs FWPF (Z1)	RT	5	0.299	0.45
		75	*a*	1.5
		175	~2.9	2.9
FWPF vs Grafoil (10 mil thick)	RT	5	<0.59	0.59
		75	1.3	1.3
		175	1.85	1.9
Ir vs FWPF	RT	5	<0.6	0.6
		75	7	7
		175	12	12
PG vs PG	RT	5	<0.9	<0.9
		75	*b*	*b*
		175	*b*	*b*
Ir vs Grafoil	RT	5	1.1	<1.1
		75	*a*	*a*
		175	*a*	*a*
	300	5	1.5	Slightly higher than RT values
		75	Slightly higher (~30%) than RT values	
		175		
CBCF-3 vs FWPF	RT	5	0.016	0.016
		75	*b*	*b*
		175	*b*	*b*

[a] Data in calculation.

[b] Dominated by insulator — contact is not important.

Notes:

1. Directional dependence of PG vs FWPF (Z1 or Z11).
2. Contact conductance of FWPF vs FWPF decreases with increasing temperature.
3. System calibrated by Cu to Cu contact.
4. Contact conductance depends on pressure history for systems involving FWPF or Grafoil.

FLIGHT SYSTEMS HARDWARE

(Activity 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 of 1255 iridium blanks and associated foil for the 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)

Production data for January are shown in Table 1. Of 216 blanks scheduled for delivery by the end of January, 173 were shipped.

Finish rolling (bare rolling to gauge thickness) of Ir sheet for blanks has been stopped while process improvements are made and a new cleaning procedure is developed. MRC has objected to the presence of microscopic inclusions in the surface of the forming blanks and will send back about 80 blanks (MRC groups P3 and P4) for removal of the inclusions. SEM work at MRC and ORNL indicates that the inclusions are primarily aluminosilicates which are presumably introduced from the fire brick muffles of the furnace used in the finish rolling process. We are setting up a new furnace which will have an Inconel muffle and heat shields so that the primary source of the inclusions — fire brick — will be completely eliminated. New tungsten carbide (WC) rolls are being installed in our Bliss 4-hi rolling mill so that the finish rolling process can be done in the Metals Processing Lab rather than on borrowed equipment at Y-12. Meanwhile we will investigate acid, alkali, and molten carbonate cleaning treatments in order to remove the aluminosilicate inclusions.

A minimum delay of one month in the delivery of forming blanks at MRC can be expected while the new cleaning procedure is developed and used to clean the 80 blanks returned by MRC and while the new equipment is installed for use in the finish rolling process. We are not considering returning to the grinding process used for MHW hardware because of major scheduling, funding, and Ir powder availability problems that would result.

Iridium Foil Production (D. E. Harasyn)

No further shipments of foil are anticipated until March. No samples of foil containing defects were received from General Electric Co. or Mound Facility.

CBCF-3 Insulation Fabrication for LWRHU (G. C. Wei, G. W. Brassell, (L. Kovach, and C. D. Montgomery)

We measured the density of six 75-mm-long CBCF-3 sleeves fabricated using perforated-plate-formed mandrel. The density and its variation is 0.20 ± 0.01 E3 kg/m^3. The load at failure of CBCF-3 full rings (16.8-mm OD x 8.87-mm ID x 6.35-mm long) is 3.3 ± 0.91 kg. CBCF-3 sleeves were also fabricated using a photoetched-foil-rolled mandrel. The density is 0.22 E3 kg/m^3. Therefore, the density of CBCF-3 sleeves is a function of the molding mandrel. Since the CBCF-3 blanks cast and formed on photoetched-foil-rolled mandrels are more uniform than those cast on perforated-plate-formed mandrel, we selected the photoetched-foil-rolled mandrel as our prime mandrel for CBCF-3 sleeve fabrication. Additional mandrels were ordered for the multiple mandrel fabrication facility.

One piece of HD CBCF-3 (0.60 E3 kg/m^3) was shipped to LASL for their dynamic tests because their earlier vibration tests on CBCF-3 plate (0.28 E3 kg/m^3) resulted in failure. We also started fabrication of HD CBCF-3 sleeves using mineral-oil-medium isostatic press.

MATERIALS TECHNOLOGY SUPPORT

(Activity AE 15 35 00 0, 189a 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 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 cause enhanced grain growth in doped Ir alloys under heat-source environments, and (4) to develop ductile high-temperature alloys acceptable for future flight missions.

Effect of Phosphorous Contamination on Impact Properties of DOP-26 (C. T. Liu)

We received four P-doped specimens (heat No. OLMF-8) from LASL for evaluation of the effect of P-contamination on impact properties of DOP-26. The four specimens were first vacuum-annealed for 18 h at 1500°C (to produce ∿14 grains/thickness) and then doped with phosphorus for 1 h at 1500°C in a graphite crucible containing a small amount of P_2O_5. Two of the as-doped specimens were impacted at 1350°C and 85 m/s (280 fps). In order to avoid a possible loss of phosphorus during heating period prior to impact tests, the specimens were rapidly heated up from 1000 to 1350°C, and kept at 1350°C for less than 2 min. The impact results are presented in Table 5 together with these from the undoped specimens annealed 19 h at 1500°C in vacuum. The undoped specimens showed ductile rupture with an average of 36% impact elongation while the P-doped specimens exhibited brittle grain-boundary fracture with 3.0–5.7% elongation. This comparison strongly indicates that the phosphorous contamination severely lowers the impact ductility of DOP-26 alloy at 1350°C.

We have previously reported that the phosphorus segregated at grain boundaries in DOP-4 Ir-0.3% W alloy can be removed by annealing at 1500°C in vacuum. Accordingly, one of the P-doped specimens was heat treated for 2 h at 1500°C in vacuum in order to remove phosphorus contamination. The vacuum-annealed specimen was then impacted at 1350°C and 85 m/s, with impact results in Table 5. This specimen showed ductile rupture with 31.6% impact elongation. The results demonstrate that the ductility of the DOP-26 specimens embrittled by phosphorus contamination can be resumed almost completely through heat treatment for 2 h at 1500°C in vacuum. Auger analyses of the P-doped specimens with and without vacuum-annealed at 1500°C are in progress.

Table 5. Effect of Phosphorus Contamination on Impact Properties of DOP-26 Alloy Tested at 1350°C and 85 m/s (280 fps)

P Contamination	Heat Treatment	Impact Ductility (%)	
		Elongation	Fracture Mode
(1) Uncontaminated	19 h/1500°C/vac.	36.0[a]	DR[a]
(2) P-Doped	18 h/1500°C/vac. + 1 h/1500°C/P	5.7	GB
(3) P-Doped	Same as (2)	3.0	GB
(4) P-Doped + Decontaminated	18 h/1500°C/vac + 1 h/1500°C/P + 2 h/1500°C/vac	31.6	DR

[a]An Average of 5 tests.

Weld Development (S. A. David)

We are continuing to evaluate laser weldability and fusion zone microstructure modification of DOP-26 alloy. As mentioned in the last monthly report of annular and crescent beam conditions, the annular beam seems to be the most appropriate to successfully weld DOP-26 alloy. Hence, arrangements have been made to make more laser welds using the laser in annular beam mode.

In view of the comments made by C. T. Bearden of Savannah River Laboratory regarding the abnormalities in Lot L DOP-26 microstructure, a critical microstructural evaluation of L-disc (heat No. L-8) welds were made compared with J-disc (heat No. JR-66) welds. The base metal grain structure of J and L discs appeared to be very similar. Both the materials are weldable and the fusion zone structure of the two welds (GTA-welds) were also found to be similar. The grain boundaries within the fusion zone were slightly decorated with precipitates presumably Ir_5Th. However, the grain boundaries within the base metal were relatively free of such precipitates. Also, no apparent grain growth or extensive precipitation along the grain boundary was observed in the base metal adjacent to the fusion line.

ISOTOPE FUEL DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 02314)

R. S. Crouse

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

The 30,000 h compatibility capsules from PNL are awaiting disassembly.

TERRESTRIAL RADIOISOTOPE APPLICATIONS DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 01367)

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

Cesium-137 Low Solubility Compounds

Two stainless steel trays were received from the shop to be used in transferring the saw for sectioning pellets and the calorimeter into the hot cell. However, after examination of the calorimeter tray it was decided that repositioning the placement pints was required to maintain the calorimeter in a stable position during transport into the hot cell. The hot cells for evaluation of the pellets will become available in mid-February.

A discussion with the HRLEL Metallography group supervisor concerning details of the electron microprobe, optical microscopy, and in-cell photography studies of the ^{137}Cs pollucite pellets to be performed was held and a procedure was written for identifying the sectioned pellets alignment and orientation with respect to the radial axis of the original cylindrical pellet.

Leach testing of the three sections of tracer level ^{137}Cs pollucite pellets continued this month. The leach rates of each pellet section are given in Table 6. The leach rate of pellet 39 decreased by a factor of 6, while the leach rates of pellets 40 and 41 remained constant to within 5% or less. This sharp decrease in the leach rate of pellet 39 is not understood at this time.

Table 6. Tracer Level Cesium-137 Pollucite Pellet Leach Rates

Pellet No.	Accumulated Leaching Period (days)	Leach Rate (g/cm^2-day)
39	210	38.4×10^{-6}
	224	6.31×10^{-6}
40	210	11.8×10^{-6}
	224	11.8×10^{-6}
41	210	23.0×10^{-6}
	224	21.9×10^{-6}

Krypton-85 Light Source Development

Six quartz spirals for use in the fabrication of light sources were prepared. Two were filled with phosphor, one yellow-green and one blue. Approximately 10 Ci of 28.7% ^{85}Kr will be loaded into each for light output

evaluation. When krypton filling techniques are developed and phosphor geometry is optimized, new spiral tubes will be filled with 48% enriched ^{85}Kr.

A reflector shield and light pipe have been completed. This will permit fabrication of a prototype light that can be used with either the blue or yellow-green light source.

Relative output measurements will be made, along with empirical measurements for distance visibility.

A batch of red phosphor has been prepared and will be evaluated after the blue and yellow-green lights are evaluated. The first light using enriched ^{85}Kr ($\sim$28%), contained a very small leak and has been unloaded and the enriched ^{85}Kr placed in storage.

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